MONTHLY PROGRESS REPORTS

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APRIL 1-30, 1988

2063

Philip Morris Research Center

Richmond, Virginia

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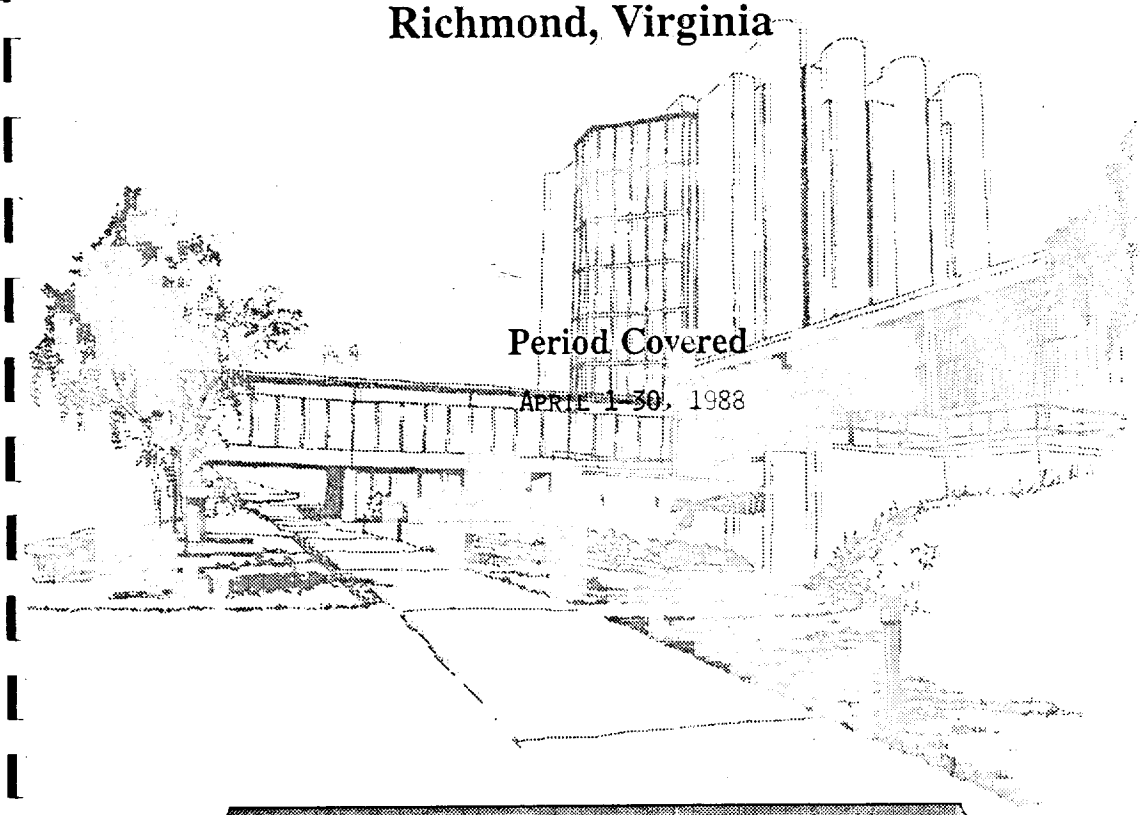

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APRIL 1-30, 1988

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*N/R = No Report

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PROJECT NUMBER: 2100
PROJECT TITLE: New Products Innovations
PROJECT LEADER: C. J. Campbell
PERIOD COVERED: April, 1988

I. PROJECT HUMIDOR

A. Objective: Develop a moisture release device for use inside a cigarette pack which will maintain the Pack OV at the desired level.

B. Status: An accelerated ageing test is in progress which incorporates a moisture control device in a menthol cigarette pack. Subjectives, OV's, and menthol deliveries are being monitored.

A contract with Klockner Packaging to produce moisture control packets has been finalized. Details for scaling up the preparation of the saturated salt solution are being discussed with Physical Research and Process Engineering.

II. PROJECT YOUTH

A. Objective: Develop a means for maintaining fresh cigarette flavor in a hermetically sealed pack.

B. Status: A test of subjectives of thermally stressed, hermetically sealed production Marlboros is in progress. If consistent subjective results are obtained (i.e., off taste and odor) then samples from the same lot of cigarettes will be irradiated and similarly tested.

III. FLAVOR ENCAPSULATION:

A. Objective: Develop the means to encapsulate flavors for use in new or improved products.

B. Status: Gelatin walled capsules made by R. T. Dodge have been evaluated under accelerated ageing and appear to retain flavors reasonably well. This will provide a more preferred material for use on a scented tear tape. A vendor capable of combining these capsules into a workable tear tape will need to be established.

IV. EMBOSSING CONCEPTS:

A. Objective: Explore the potential advantages of embossing cigarette paper, tipping and plug wrap for both cost savings and aesthetic appeal.

B. Status: The embossing equipment in the lab is being modified to provide additional capabilities.

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V. PACKAGING CONCEPTS:

A. Objective: Develop novel package designs.

B. Status: Discussions have been held with two new vendors regarding their capability to mold a new style plastic pack.

VI. TAMPER EVIDENT PACKAGING:

A. Objective: Develop packaging materials and processes for obviously indicating an attempt to tamper with a pack.

B. Status: Concept samples of packages with tamper evident features were presented to New York management for their review. The status of the work on developing a puncture evident film was also presented.

A two day industry meeting on the latest developments of tamper evident packaging was attended by Chris Campbell.

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PROJECT NUMBER: 2105
PROJECT TITLE: Filter Development
PROJECT LEADER: W. A. Nichols
PERIOD COVERED: April, 1988

I. FILTER MATERIALS, LTD.

A. **Objective**: Develop polypropylene tow that is equivalent to cellulose acetate.

B. **Results**: Samples requested by TTG for International products were produced.

Evaluation of the wax application system was begun on the KDF-2. Samples have been produced with wax at 300m/min. for short durations. The heating system used to hold the wax in a liquid phase is being tuned to maintain operating temperature.

C. **Plans**: Operating conditions for the wax application system will be established.

II. NEW FILTER CONCEPTS

A. **Objective**: Develop unique products using novel filters.

B. **Results**:

Modified Plastic Filter: Processing inefficiency was studied and found to be attributed to filter length variation. Since the last supply of filters, Louisville installed a more accurate filter cutter. Filters have been made and are in transit. Samples will be made for E.E.M.A. as soon as the filters are received.

PVA Matrix: Cigarettes were produced to evaluate supplementing menthol on foil with PVA Matrix containing menthol. An aging study is in progress and samples will be smoked for subjectives.

Concentric Filters: Concentric filters having a PVA Matrix with menthol coated on the outside of the center core were received. As the Filtrona process uses steam application for fiber bonding, there were concerns that the menthol would be volatilized. Analysis of menthol indicated little loss. Cigarettes are scheduled to be fabricated.

C. **Plans**: Previous Modified Plastic Filter processing has been done with continuous adhesive application; patterned application will also be evaluated. The PVA Matrix aging study will be completed in May.

III. MENTHOL ON FOIL

A. **Objective**: Support the introduction of menthol on foil process.

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B. **Status**: Construction of a third menthol on foil applicator nears completion. This machine will be tested and shipped to Louisville. One of Louisville's existing machines will be returned and upgraded.

C. **Plans**: Produce samples as requested.

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PROJECT NUMBER: 2304
PROJECT TITLE: Flavor Development - New Products
PROJECT LEADER: Barbara G. Taylor
PERIOD COVERED: April, 1988

I. FILTER ADDITIVES

A. **Objective**: A. Evaluate the effects of filter additives on the subjective characteristics of cigarette smoke (evaluations on blends and blend components. B. Use filter additives to modify and enhance the most desirable smoke characteristics.

B. **Status**: GCC Marlboro cigarette models were made using filters with triacetin (8%) and citric acid at various levels (0.0, 1.95, 2.85 and 3.10 mg) via dual application. In another model, glycerine was applied to prevent the delivery roller from gumming up. The analytical results show a significant reduction in the nicotine delivery of the test models with citric acid. Accelerated aging studies have been initiated to evaluate the effects on nicotine delivery.

Investigations of alternate PZ's are under way to eliminate the need for a dual application of the citric acid. Triethyl citrate (TEC) and Carbowax 750 were being considered, however, Carbowax 750 has machineability problems. The use of TEC is being evaluated further.

II. FOAM BOUND RODS

A. **Objective**: To achieve reduced tobacco weights in PM products via use of the foam bound technology.

B. **Status**: Subjective results have been completed on the models with Star Dri maltodextrin. The results show significant differences between the test and control. Models from run 147 using OBI apple pectin have been subjectively evaluated, the results are being tabulated.

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PROJECT NUMBER: 2305
PROJECT TITLE: Flavor Development-Brand Modification-Menthol/Distinctive
PROJECT LEADER: H. M. Maxwell
PERIOD COVERED: April, 1988

I. ALTERNATIVE FLAVORS:

A. Objective: To identify and qualify alternative sources for flavoring materials.

B. Status: An alternate vendor for block licorice is currently being considered. Semi-works cigarettes are being produced for the Cross-Operations panel. Another casing flavor alternative is being tested for qualification.

C. Plans:

Cigarette Remake	April, 1988
Cross Operation Mail-Out Panel Results	May, 1988
Cigarette Production For Casing Flavor POL (Merit 85)	May, 1988

II. PROJECT MOOG:

A. Objective: To develop the expertise to produce cigarettes that are subjectively equivalent to Salem, Newport and Kool.

B. Status: The N-type POL results indicate that MOOG-N is similar to Newport. Flavor modification for Moog K and S will be initiated upon receipt of suitable models from Semi-works.

C. Plans:

K-type Semi-works Cigarettes	May, 1988
S-type Semi-works Cigarettes	May, 1988

III. PROJECT LEVO:

A. Objective: To determine the subjective differences between 100% Natural and 100% Synthetic menthol and to develop flavors to make them subjectively equivalent so that manufacturing would have complete flexibility to use them at any ratio. Also, to qualify alternate suppliers and/or manufacturing processes modifications.

B. Status: Cigarettes for Merit POL retesting of 100% natural vs. 100% synthetic menthol are approved for testing. In-house testing

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and a B&H Menthol 100 POL is also scheduled. An alternate supplier and a manufacturing process modification by a present supplier was qualified.

C. **Plans**:

POL Results	June, 1988
B&H POL	To Be Scheduled When Smokers Are Available

IV. **MENTHOL RELEASE COMPOUND**:

A. **Objective**: To develop a mentholated charcoal-filtered cigarette utilizing a menthol release compound.

B. **Status**: Additional cigarettes will be made utilizing the foam bound rod process to apply MGC. These cigarettes will be used to compare menthol delivery characteristics of MGC with CA filter, charcoal in tow filter, and charcoal in plug-space-plug filter.

C. **Plans**:

Additional Cigarette Production	May, 1988

V. **PROJECT GRAIN**:

A. **Objective**: To significantly reduce alcohol levels in PM flavor systems while maintaining product integrity.

B. **Status**: A run at Stockton Street will be scheduled to produce cigarettes with 0, 10, and 30 percent alcohol reduction.

C. **Plans**:

Stockton Street Run	June, 1988

VI. **ALTERNATE HUMECTANT PROGRAM**:

A. **Objective**: To develop the capability to produce cigarettes that are glycerin-free and cigarettes that are propylene glycol and glycerin-free, while maintaining subjective integrity.

B. **Status**: G-free cigarettes were made and are being evaluated for POL testing. Casings and aftercut were made replacing PG and G for that program.

C. **Plans**:

G-free POL Release	April, 1988

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Semi-works PG-G Free Cigarette Production — June, 1988

VII. **PROJECT FAMOUS**:

A. **Objective**: To optimize US export Chesterfield, and from this basis define guideline specifications for Chesterfield worldwide.

B. **Status**: Two prototypes have been developed on-site at FTR. They are in consumer testing in Switzerland. Translation of the prototypes from FTR to Semi-works manufacture is progressing.

C. **Plans**:

Semi-works Optimization	Ongoing
Continued Product Development	Ongoing

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PROJECT NUMBER: 2306
PROJECT TITLE: Marlboro Standardization and International Support
PROJECT LEADER: J. L. Spruill
PERIOD COVERED: April, 1988

I. MARLBORO STANDARDIZATION

A. Objective: Analytical and subjective evaluations of production Marlboro KS/LS.

B. Status: A March factory pick-up is under evaluation analytically and subjectively.

C. Plans: Complete March factory pick-up evaluations.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

A. Objective: To provide subjective direction for programs within R&D and manufacturing locations.

B. Status: Twenty panels completed during the reporting period. Additionally, Menthol Aging study samples evaluated. Profiled eight brands from competitive companies.

C. Plans: Provide assistance as needed.

III. PROJECT NATURAL

A. Objective: To develop 85 mm full-flavored and lights prototypes using blend components and flavor systems which will provide a natural blended product.

B. Status: Ad pack test samples have been made and are being subjectively and analytically evaluated. Six POL tests have been requested (three 85 mm length, three 100 mm length) with single-digit tars using Virginia, Virginia/Oriental and Blended blends for testing against each other. A 5,000-pound trial run of the Virginia/Oriental prototype will be made at Stockton Street the week of April 25th to iron out possible problems before production run in early May. Mentholated foil samples will be run at the same time.

C. Plans: Fabricate POL samples and evaluate Stockton Street trial cigarettes.

IV. FLAVOR RESOURCE DATA BANK

A. Objective: Creation, customization and maintenance of flavor resource and data files for use by Flavor Development Division.

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B. **Status**: Vendor samples being evaluated.

C. **Plans**: Division updated with information.

V. INTERNATIONAL SUPPORT

International Brands Smoking Panel

A. **Objective**: Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.

B. **Status**: Eleven panels completed as well as evaluations of production Larks, L&M Milds and Manhattan 85's and 100's. Hi-Lite Milds and New Paradise profiled.

C. **Plans**: Provide assistance as needed.

PROJECT STARSHIP

A. **Objective**: Develop a 12 mg Chesterfield for Japan.

B. **Status**: Evaluating L&M Milds production cigarettes.

PROJECT "DANNY"

A. **Objective**: To develop a cigarette for production in Malaysia with inclusion of up to 50% local tobacco subjectively comparable to U.S. Marlboro.

B. **Status**: Evaluated production samples from Malaysia. Decision will be made if flavor modification is required.

PROJECT LIGHT/ULTRA

A. **Objective**: Develop cigarette models at 4, 6 and 8 mg with a new blend and flavor system.

B. **Status**: Cigarette models under subjective evaluation with possible models for Danchi Testing.

C. **Plans**: Continue flavor development if needed.

PROJECT SWEET

A. **Objective**: Development of a distinctively sweet cigarette for the Japanese market.

B. **Status**: Subjective evaluations of "sweet" models still underway - possible release for Danchi Testing.

2000459524

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PROJECT MOUNT ULTRA-JAPAN

A. **Objective**: Explore the use of new blends, new flavor systems and different construction styles. Cigarettes will be in the low and ultra-low category for the Japanese market.

B. **Status**: 85 mm cigarettes were made with P.M. Super Lights cut filler. Various cigarette papers were used, as were filters of different lengths. Of the cigarettes screened, we felt that subjectively, the following models warranted further subjective evaluation:

PACK CODE	FILTER LENGTH	TAR	CIGARETTE PAPER
5939	25 mm	4.6 mg	664
5945	27 mm	5.7 mg	P1166
5941	21 mm	6.2 mg	611
5942	21 mm	6.2 mg	P1166

C. **Plans**: Further models being requested using flavors developed for Project Light/Ultra (primary processing 4/27). Upon receipt of various cigarette models, further aftercut systems (including those used in Low Tar/High Taste) will be evaluated.

2000459525

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PROJECT NUMBER: 2307
PROJECT TITLE: Flavor Investigation/Nonvolatile Flavor Investigation/Processed Tobacco
PROJECT LEADER: W. R. Raymond
PERIOD COVERED: April, 1988

I. FLAVOR INVESTIGATION:

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Results:

1. Project Grain--Repeat analyses of ethanol and multidimensional GC analyses of two major volatile constituents were completed for control versus sonulated A/C formulations with 30% and 100% ethanol replacement levels. Analytical ethanol values were consistent with formulae levels. However, significant differences in the two volatile components were observed among samples.

2. Marlboro POL's--Complete flavor analyses (A/C's, A/C concentrates and casings) were performed for POL 3584 and 3585 production runs at Stockton Street on 4/18/88. Also determined were A/C levels on filler. Analytical results indicated conformation to formula specifications and correct A/C application levels.

3. Menthol--A time study was completed to monitor residual ethanol levels in foil- vs spray-mentholated production cigarettes of known history. Two analytical methods, an extraction method and a headspace GC method, were used in parallel and a strong correlation established between the two. The residual ethanol level in B&H Menthol (spray application) cigarettes was ca. 200 µg/cigarette initially and exhibited negligible change through four weeks of ambient temperature storage. Of three foil-mentholated packings studied, all had initial ethanol levels in excess of 1000 µg/cigarette, two exhibited only ca. 20% decreases in ethanol after the fourth week of storage and the third, MFLM 100's FTB, exhibited a ca. 70% decrease during the same period. The need for further studies is indicated.

4. TRIM--Complete analyses were performed on TRIM flavors and casings. With the exception of the PG level in the Burley spray, all values were within specified ranges. Samples of two proposed production tipping adhesives for TRIM were analyzed by headspace GC to try to determine possible causes for subjective differences. No major volatile solvent differences were noted.

5. Analytical Method Development--A new GC procedure was developed to quantitatively monitor volatile components in Burley top casing for specification and Q.A. purposes. A Q.A.

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procedure was prepared for "Determination of Glucose, Fructose, Sucrose and Glycerin in Flavor Materials by HPLC."

6. Applied Flavor Investigation--Based on frequent encounters with acetal formation in PM flavor systems, PG and glycerin acetals of eight commonly-used aldehydes were prepared and analyzed by GC and GC/MS to obtain retention data and reference spectra.

7. Miscellaneous--GC profiles for Merit and Half-Pint A/C's were determined and compared to prior profiles. Two revision flavors were compared by GC versus prerevision retain samples and purities were determined for two flavor chemicals. Headspace GC/MS analyses were performed on several samples of packaging materials including two types of promotional sleeves with U.V. cured printing inks.

II. NONVOLATILE FLAVOR INVESTIGATION:

A. Objective: To investigate tobacco-natural, tobacco-identical and other nonvolatile fractions and constituents for potential application as cigarette flavor additives.

B. Results:

Samples of subcuts from Bright tobacco extract carbon fractions III and IV were received from S. Tafur for subjective evaluation. Evaluation is in progress.

III. PROCESSED TOBACCO:

A. Objective: To develop basic and applied knowledge for the purpose of improvement or selective modification of subjective properties of processed tobaccos.

B. Results:

1. RLB--Modified dry flavor replacement samples were requested from one supplier for analytical and subjective comparisons with material originally submitted.

2. ART-Stem--Post-ART CRS from trials 96-101 is being evaluated for direct utilization in Marlboro and PMSL. Subjective testing of post ART IS vs. IS, both at 5% in MF, indicated a tendency toward reduced liking scores for the test IS. In PMSL, the control IS also had higher liking scores. In MF, the test IS was perceived as dryer, more dirty and earthy. In PMSL, the control was perceived as smoother, less hot and less peppery then the post ART material.

3. Stems--Marlboro cigarettes containing cased IS versus ES have been submitted for analytical and subjective comparisons prior to release for POL tests.

2000459527

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PROJECT NUMBER: 4009
PROJECT TITLE: Smoke Studies
PROJECT LEADER: B. L. Goodman
PERIOD COVERED: April, 1988

I. PROJECT STUDIO (R. Slagle)

A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream visibility.

B. Results: A final prototype in the low sidestream Trim configuration has been selected:

1. 10-046-A Wrapper - 4.7% K-Succinate, 0.7% NaCMC, perforated to 50 Coresta.
2. Trim II Blend/-49 Aftercut System
3. Filler cut to 40 CPI

Development with DMCP and Engineering has resulted in the capability of DMCP to electrostatically perforate the special $CaCO_3$ cigarette paper bobbins for production in test market quantities.

Further work continues to analytically and subjectively optimize a low sidestream candidate, in the following directions:

1. Development of a reduced level $Mg(OH)_2$ Ecusta wrapper, possibly incorporating a lower basis weight.

2. Further evaluation of chemical additives (e.g., MAP) in the KC small particle size $CaCO_3$ paper.

Factory panel testing of Capri versus three low sidestream Trim models showed no significant difference between Capri and two of the models:

	Acceptability
1. 2123-42(PERF)	4.17
2. 2131-41	4.13
3. Capri	4.07

However, the third model (2123-42 Unperforated) was significantly lower (3.92) in acceptability. An internal mailout of a low sidestream candidate (2123-41) and Trim I showed no significant difference (3.95 vs. 3.83).

A 100 mm model with the low sidestream $Mg(OH)_2$ paper was tested on M/C panel versus a control Marlboro Lights 100 (unidentified). There were significant differences in all attributes with acceptability scores of 4.23 for the control and 4.03 for the test. No further testing of a 100 mm cigarette is planned at this time.

2000459528

Source: https://www.industrydocuments.ucsf.edu/docs/nfm0000

Porous cigarette paper was coated with a 5% solution of water soluble cellulose acetate with and without a small quantity of added $CaCO_3$. The additives were suggested by G. Keritsis as a way to smooth the smoke and to get some sidestream reduction by melting of coating around the charline. The sidestream visibility for the coated model with $CaCO_3$ was decreased by 13% and for the WSCA the reduction was 22%. The coatings made the papers nonporous (55-90 seconds Greiner), which caused mainstream deliveries to increase correspondingly. Preliminary subjective smoking indicated that the coatings might have given the desired smoothing effect compared to what would be expected for non-porous papers. It is questionable whether the coatings contributed to any additional reduction of the SS visibility beyond the effect of the porosity change.

Sidestream visibilities were measured for many different versions of the small particle size $CaCO_3$ paper. Higher reductions were seen for the cigarettes made in Louisville than previous models that had been less densely packed with tobacco. All four K-C production papers gave visibility reductions around 58% compared to Capri controls. Perforation of the wrapper did not significantly change the visibility reduction. MAP on the K-C paper gave a 40% reduction, while the Trim I paper gave 12% lower SS visibility than Capri.

C. **Plans**: Continue investigation of chemical loading on the $CaCO_3$ paper, and development of reduced level $Mg(OH)_2$ wrappers.

II. TIPPING PAPER INVESTIGATIONS

A. **Objective**: To examine different papers for appearance, composition and performance.

B. **Results**: A series of cigarettes were made with black or white tow on the inner section of a charcoal filter. Each tow combination was wrapped with three different plug wraps and two different tippings. Selected models were submitted for O/C panel testing of appearance, specifically at the filter junction.

The white tow models were rated as having less obvious junctions than the black ones. A nonporous perforated plug wrap concealed the junction better than the porous 33M1, with a higher opacity porous plug wrap falling in the middle. The difference between a higher basis weight and a normal basis weight tipping was not significant.

C. **Plans**: Determine the variability of dilution on cigarettes with the perforated plug wrap.

Compare previous combinations with the current ones.

2000459529

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PROJECT NUMBER: 2304, 2305, 4015
PROJECT TITLE: New Product Development
PROJECT LEADER: B. G. Taylor, H. Maxwell, and W. G. Houck
PERIOD COVERED: April, 1988

I. MARLBORO/MARLBORO LIGHTS MENTHOL

A. Objective: (1) Develop Marlboro Menthol to appeal more to Salem smokers. (2) Develop a Marlboro Lights Menthol brand extension to appeal to Salem Lights smokers.

B. Status: National retail sales in progress since 2/1/88. Menthol monitoring continues.

Replicate POL and HTI testing of Marlboro LTS Menthol vs Salem LTS now scheduled for shipment the week of 5/2/88 (POL tests 2148/49)

Phase II testing of techniques to stabilize menthol deliveries during aging is **currently in progress.** This testing includes "heat" setting of menthol in cellulose acetate and supplemental release of menthol via PVA menthol encapsulations. Initial information from this testing available mid-May

C. Plans:

Complete replicate POL's 2148-49 and HTI 1719	6/88
Monitor National Prod. Start-up	On-going
Special Studies (Initial Info)	On-going

II. MARLBORO ULTRA LIGHTS

A. Objective: To develop 85/100 mm Ultra Low (6 mg) candidates for Marlboro line extensions.

B. Status: POL testing scheduled versus MUL 85 and 100. Cigarette making scheduled to be completed the week of 5/2/88.

C. Plans:

Complete POL testing vs MUL	6/88
Test Market	4th Qtr.'88

III. PROJECT TRIM (Ultra Slim)

A. Objective: To develop 17 mm circumference/100 mm Ultra Slim product candidates.

2000459530

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B. **Status**:

TRIM I:

POL testing of mentholated foil product completed with satisfactory results.

POL testing of Louisville produced Product vs Capri scheduled for both regular and menthol prototypes. Cigarettes prod tipping and hand-packing scheduled for the week of 5/2/88.

C TRIM IV: (Low Sidestream)

Subjective satisfactory cigarettes were produced in Louisville the week of 4/18/88. Tip to rod air leaks, however, are a problem with the specified National 1979 tipping adhesives. Alternate adhesives are currently under evaluation from both air leak and subjective view points. Currently, Findley D42481 and Fuller 3924 adhesives look promising as a qualified alternates to the national 1979 adhesive.

Regular and menthol POL cigarettes for TRIM I vs TRIM IV testing will be made in Louisville with the Findley adhesive 5/2/88. Test market salesman samples are now scheduled to be in place on June 20th, 1988.

C. **Plans**:

Louisville Factory Trials	Completed
TRIM POL's vs Capri (Make cigarettes in Louisville)	4/27/88
TRIM I vs TRIM IV POL's (Make cigarettes in Louisville)	5/2-5
Test Market Specification	By 5/5
Factory start-up	5/9
Salesman Samples (TRIM I or TRIM IV at sales meeting)	6/20
Test Market (retail)	8/88

IV. **PROJECT ART**

A. **Objective**: To investigate methods to decrease the nicotine content in tobacco. To develop full-flavored, ultra low, and light products using the low nicotine tobacco products.

B. **Status**: Refined prototypes at 6,8,11, and 16 mg tar using the "DL" blend have been made and are under analytical and subjective evaluations.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

Internal extended use testing vs Marlboro Lights and Carlton is planned using an 11 mg ART model with the "DL" blend.

Product Development will continue to concentrate on "Merit" and "Marlboro LTS" direction as well as a "free-standing" menthol.

C. **Plans**:

Product/Flavor Development	On-going
Internal Testing	2nd Qtr. '88
Test Market	1st Qtr. '89

V. PROJECT NATURAL

A. **Objective**:

Phase I

To develop "All Natural" cigarette candidates with no additives to the tobacco.

B. **Status**: Ad/pack tests of the 11.5 mg/1.12 mg nicotine model ready for shipment. These are semi-works produced cigarettes with two different labels. One label carries the term "All Natural Cigarettes", while the other adds the following statement: No additives, no preservatives, no artificial flavor, just 100% natural tobacco.

A Factory Trial was completed on 4/27/88 at the Stockton Street Factory with satisfactory results. Cigarettes were made at 11.5 and 13.0 mg tar in both regular and menthol.

C. **Plans**:

Ship Ad/pack	4/29/88
Select Final Product Tar level (either 11.5 ro 13.0 mg)	week of 5/2/88
Possible second Factory Trial	week of 5/2/88
Factory start-up	5/16/88

VI. PROJECT EXTRA

A. **Objective**: The development of 4-8 mg product candidates that have the subjective taste of products with twice the tar.

B. **Status**: Six samples of all lamina blend were cased and will be made into cigarettes using the low density rod technology. Casings included Marlboro, Half, Pint, TBC 8544-43, casing 8544-56, casing 8493-188A, and casing 7367-115.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

Models were completed the week of April 25 to evaluate paper filter components from Baumgardner Papiers and Intertaba. Gas phase analysis is complete for cigarettes from POL test 0125 (CA filter) and 0126 (paper/CA).

CR chemicals continue to be subjectively evaluated. CR 2673 - Glucovanillin - is presently under evaluation as a vanillin release compound. CR 2643 - Coumarin substitute - is presently under consideration for phase III. Several new casings and aftercuts have been developed and are being used in Projects Art and Trim.

Results of the 9th month storage study of Super Juice II indicate little change in the organic components at room temperature. Subjective smokings of the 9-month old product vs. freshly made product show no significant difference in aromatic profiles.

C. **Plans**:

Paper Filter Evaluations	In progress
Sensory Mapping	On-going
Flavor development/screening	On-going
Prototype Development/evaluation	On-going

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

PROJECT NUMBER: 4022
PROJECT TITLE: International Product Development
PROJECT LEADER: L. Mims Evans, R. E. Tinker
PERIOD COVERED: April, 1988
WRITTEN BY: A. H. Confer

I. PROJECT DORA (HONG KONG)

A. Objective: Develop an 11.5 mg KS product in both charcoal and non-charcoal versions.

B. Results: Initial production of 16 million units was accomplished at Stockton Street the third week of April.

C. Plans: Cigarettes will be shipped for a June launch as Manhattan KS and 100.

III. PROJECT DRAGON (PRC)

A. Objective: Develop a "blended" KS product at 17-19 mg. tar. The product is to be a non-PM trademark, owned and manufactured by CNTC (China National Tobacco Corporation).

B. Results: Flavor concentrates have been prepared, analyticals performed and costed. Three blends have also been prepared and costed. The PM USA/PM Asia team visited the Zhangjiakou factory to assess its manufacturing capabilities for producing this cigarette.

C. Plans: Submit costs to the CNTC via Hong Kong management to obtain a go/no-go decision from Chinese.

IV. PROJECT DANNY (MALAYSIA)

A. Objective: Increase inclusion of local tobacco in Marlboro while attempting to maintain taste character.

B. Results: A 50% local tobacco inclusion prototype has been locally manufactured and analytically and subjectively approved in Richmond.

C. Plans: Consumer test in Malaysia the prototype vs the 35% local inclusion control. A 40% local inclusion model is also being developed.

II. PROJECT FAMOUS (VARIOUS COUNTRIES)

A. Objective: Optimize Chesterfield as a worldwide competitor to Camel.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

B. **Results**: Domestic testing of Model 44P, Chesterfield Export KS, Camel KS, and the French Camel KS and LS is in progress on the non-menthol mapping panel. Consumer testing of three models vs the French Camel KS is not yet finished.

C. **Plans**: Select prototype(s) based upon results of both consumer tests.

CONFIDENTIAL

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

PROJECT NUMBER: 4024
PROJECT TITLE: Japan Product Development
PROJECT LEADER: C. B. Altizer
PERIOD COVERED: March, 1988

I. LARK

A. Objective: To maintain the subjective acceptbility of the Lark family while retaining the American blended cigarette character.

B. Results: Results were received from the Danchi Test of Lark Milds evaluating expanded tobacco/grade substitutions. The test showed no statistically significant difference in liking scores for the model using BLDET in place of LET. Results from the PMI test evaluating the same change are still pending and should be available by late May.

C. Plans: Plans for implementing the expanded tobacco substitution will be finalized based on PMI testing results.

II. PROJECT STARSHIP

A. Objective: To develop a product in conjunction with a Young American Image, that would compete successfully with the latest generation of U.S. imports.

B. Results: L&M Milds (Starship L&M Prototype) was launched in Japan April 1. Two PMI Tests, L&M Milds vs Viceroy Milds and L&M Milds vs Islands, were prepared for testing that will begin May 12, 1988. A PMI test, L&M Milds vs HiLite Milds has been scheduled for June 1988. These PMI tests should assess how well we have addressed the value segment of the Japan Market.

C. Plans: Further work for project Starship will be based on the results of the PMI testing.

III. PROJECT MARLBORO

A. Objective: To determine if Marlboro Japan consumer acceptability would increase with the use of Marlboro Lights Japan blend and/or a charcoal filter.

B. Results: A PMI test was prepared and shipped for Marlboro USA vs Marlboro Japan. Results of this test are expected by Mid-June. Marlboro Japan and Marlboro Lights Japan cut filler were received from J.T.I. This filler will be used to prepare cigarettes for Danchi Panel testing of current Japan Marlboro and Marlboro Japan with Marlboro Lights filler and a charcoal filter.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

C. **Plans**: Based on results of the Danchi testing a new Marlboro Japan will be specified for an additonal Danchi Panel test. The new Marlboro Japan will be tested versus the current Marlboro Japan, Marlboro U.S. and Lark full flavor.

IV. PROJECT LIGHT ULTRA

A. **Objective**: To expand product capability in the low and ultra low tar areas.

B. **Results**: Two prototypes were developed at 6 mg. tar delivery. Subjective evaluation, versus Mild Seven and Mild Seven Lights, by the Richmond Panel indicated that these prototypes are ready for Danchi Panel testing. Prototypes at 3 to 4 mgs were designed and requested.

C. **Plans**: If subjective evaluations of the 3-4 mg. prototypes is favorable these prototypes will be tested on the Danchi Panel.

V. PROJECT MOUNT ULTRA

A. **Purpose**: To develop Mount prototypes in the ultra low tar range as a possible line extension for the Philip Morris Product Family.

B. **Results**: Prototypes were developed at 4.5 mgs and 6.0 mgs. Subjective evaluation by the Richmond Panel indicated that the prototypes needed more hotness and impact.

C. **Plans**: Develop prototypes at 3-4 mgs with improved subjective response.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

PROJECT NUMBER: 5001
PROJECT TITLE: Packaging Studies
PROJECT LEADER: H. R. Dunaway
PERIOD COVERED: April, 1988

PACKAGING STUDIES

A. **Objective**: Assist New Products Directorate in evaluating new packaging concepts and products, and provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance.

B. **Status/Plans**:

1. **Project Fox**: Four box samples with various color pigments, submitted by Nypro using their most promising resin (#55500), have been analyzed by Headspace/GC and subjectively evaluated. Based on results from these and previous tests, we feel that this plastic pack will be subjectively acceptable, for use in consumer testing, if heat treated prior to packing.

2. **Polypropylene Laminated Blanks and Cartons**: A study has been initiated to evaluate flip-top boxes and cartons, with poly laminated to the inside surface, for improved moisture retention. Currently awaiting adhesive samples for machine testing.

3. **Flavor Development Menthol Aging Study**: Assisting in evaluating 20 menthol brands by providing: OV's on tobacco, labels, FTB's, and cartons; cigarette spotting and staining comparisons; WVTR and gauge of pack wraps; and menthol absorption rates of packaging materials.

4. **Residual Printing Solvents in Packaging Materials**: Coordinated testing and advised Purchasing on the acceptability for use of the following items in production:

- Elan 100 Reg & Men FT 20, FT 20 COMP, FT 20/200
- Elan Reg & Men Tipping Paper
- Toluol Free Marlboro Tipping Paper
- Manhattan 100 SP 20, 20/200, KS FT 6
- All Natural KS & 100 SP 20
- All Natural Tipping Papers
- PM Super LTS FT 6 (European and Domestic)
- Alpine SBS Promo Materials
- Alpine 2 for 1 Poly Band
- Alpine Pack Coupons
- Alpine 25¢ Off Pack Sticker
- Merit Taste Challenge '88 Pack Sticker

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

PROJECT NUMBER: 0400
PROJECT TITLE: Low Density Rod Development
PROJECT LEADER: R. S. Mullins
PERIOD COVERED: April, 1988

LOW DENSITY ROD

A. **Objective**: Develop a continuous process for the production of reduced density cigarettes.

B. **Results**: Relocation of the vacuum pump to D125 was completed. The addition of the second pump appears to have significantly improved the performance of the tipper at higher speeds. Prior to the addition of the second pump, tipping performance would begin to deteriorate at a speed of about 1250 cpm because the vacuum was not sufficient to hold the components securely on the drums at that speed. Following the relocation, the maker and tipper were operated at a speed of 2000 cpm. A five minute run was made during which cigarettes were made at a weight reduction of 25% (800 mg total weight) and an OV of 13% to 14%. The run was stopped by a jam on the tipper rolling drum. The maker is being set up to operate at a speed of 1000 cpm for normal sample production as this is the highest speed at which the wrapped rod can consistently be manually started through the cutter head.

Two baseline runs were made to evaluate the effect of binder solution concentration (2% vs 6%) on cigarette quality using cased all-lamina filler. This cased filler ran very well, producing some of the best cigarettes that have been made in the low density process. However, as had been seen on previous runs using uncased filler, the material coated with the 6% concentration solution appeared to produce lower quality cigarettes than that coated with the normal 2% concentration even though the binder addition level was the same for both fillers (6% by weight). Four additional runs will be made to provide enough data to allow the effect of the binder concentration on the performance of the cased filler to be statistically evaluated.

Production of cigarettes for both the CV/OV/Size test and the preliminary casing evaluation were completed and samples submitted for testing. Both physical and analytical data from the Product Designs Study has been received and is being analyzed.

C. **Plans**: Continue investigating the effect of binder solution concentration on the physical quality of low density cigarettes. Continue preliminary evaluations of binder application to filler at the maker. Analyze the results of the Product Designs Study, the preliminary casing evaluation, and the CV/OV/Size test.

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PROJECT NUMBER: 1307
PROJECT TITLE: Reconstituted Tobacco Development
PROJECT LEADER: R. G. Uhl
PERIOD COVERED: April, 1988

I. IMPROVED SHEET PROPERTIES

A. Objective: Improve the physical characteristics and blend performance of reconstituted sheet materials.

B. Results:

1. ART Project - Subjective screening of RL sheets incorporating ART stems shows a subjective preference for shredded stems over CRS. However, post-ART CRS made into IS at Louisville and subsequently incorporated into RL gave the least objectionable subjective response to date.

   A second study of cigarette-to-cigarette blend variation for stem components (ES, IS, shredded stem) confirmed that variance increases with inclusion level but is independent of stem product type.

   Short term microbial stability studies on pre and post-ART stems show no obvious differences. Statistical verification will be difficult due to variation in results, even between duplicate samples. Long term stability studies are in progress.

2. Humectants - Control and test cigarettes for POL evaluation of glycerin-free blends were subjectively screened by the Richmond panel and deemed subjectively unacceptable. Casing formulations will be adjusted and POL cigarettes remade.

   PG/glycerin-free RCB was made at the BL Plant to complement the RL products made at Park 500 in March. The test RCB was targeted at 2% lower packout OV (compensation for the absence of a volatile PG component), but had to be run at normal OV levels to eliminate cracking. The test sheet was also less uniform (thick/thin spots). This material is nevertheless acceptable for use in blends to evaluate PG/glycerin-free strip casings and aftercuts.

   Oven OV values derived by the PM-USA method (100°C for 3 hrs) vs the PME method (103°C for 100 minutes) were compared using sheet components and MKS blend. PM-USA would report a higher OV value (Δ = +0.3). This difference would be reduced to 0.2 without RCB (Δ OV for RCB = +0.7 by PM-USA method vs PME method).

   Pilot RL sheets containing individual humectants and no humectant are being produced for Semiworks processing (at several OV levels) to compare the effectiveness of humectants vs water in improving survivability.

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3. RL - Licorice incorporated into pilot RL baseweb showed no improvement in baseweb absorption of size, either visually or by droplet contact angle measurement. Glycyrrhizic acid analysis of baseweb showed licorice is preferentially retained by the baseweb on the paper machine.

C. **Plans**:

1. Produce pilot RL to subjectively evaluate ART stems at reduced monopotassium citrate levels.

2. Evaluate ART stems at the final citrate level to determine an incorporation rate that maintains subjective parity in RL.

C

II. SUBJECTIVE MODIFICATION OF RL

A. **Objective**: Improve or modify the subjective character of RL.

B. **Results**:

1. Analysis over the life of Park 500 150B size batches (from fresh to thickening/recycle) showed no change in the ratio of analyzeable flavor components. Insoluble solids increase by half; this is attributed to baseweb fiber and possibly accumulation of dry flavors rejected at the size press nip. Viscosity doubles and there is a threefold increase in yield stress (force required to fluidize the gel). Thickening increases size application to the baseweb, and hampers control of finished sheet solubles level.

2. Subjective screening of dry flavor replacements (would allow size screening and retard or eliminate thickening) has shown two acceptable suppliers for each of the two replacement flavor components. Native extracts (intermediate products) of the liquid flavor from both vendors are being analyzed in an effort to attain interchangeability and a common analytical specification for both suppliers. Shelf life of the liquid flavors will also be evaluated.

3. A nondisclosure agreement was sent to Branson Sonic Power prior to renting an ultrasound unit for evaluation on breaking up Modified 150B size precipitates. The pilot rental unit should be available at the end of May. Other groups have requested testing time to evaluate its applicability to pectin binder extraction/mixing and binder defoaming.

C. **Plans**:

1. Produce pilot sheets to evaluate liquid flavors for batch to batch consistency and shelf life.

2. Complete the piping design and installation to evaluate an ultrasound unit in the pilot size press system.

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PROJECT NUMBER: 1313
PROJECT TITLE: Semiworks Process Development
PROJECT LEADER: W. G. Sanderson
PERIOD COVERED: April, 1988

A. **Objective**: Provide engineering and technical support to improve the performance and efficiency of the Semiworks operation.

B. **Results**:

Bright Casing Application System - Semiworks Primary (Nguyen) - The installation of a second bright casing application system was completed. This system, which is identical to the original bright casing application system, will be used primarily as a standby unit. The availability of a second system will provide improved flexibility for bright casing application and reduced downtime between the application of dissimilar flavors.

Aftercut Flavor Application - Semiworks Primary (Nguyen) - A manual detailing the operating procedures for the aftercut flavor application system was issued. This manual contains operating standards, flow meter calibration procedures, cleaning requirements, system lay-out diagrams, and control logic schematics. Detailed operating procedures for training purposes are also included.

Optical Inspection System - Dual Ring Tipper (Osmalov) - P.M. U.S.A. Engineering presented a proposal for the installation of an optical inspection system for the dual ring tipper in Semiworks make/pack. Mechanical design work is in progress for the double rolling of the tipped cigarette (to allow the optical inspector a full 360 degree view of the cigarette) and for a mechanism to eject defective product. Engineering expects to have the mechanical components designed, fabricated, and evaluated on the machine by the end of July, 1988.

C. **Plans**: Continue with the preparations for the mixing cylinder/second aftercut flavor application system conversion. Transfer the technology developed for the TLA primary process to the Semiworks small scale primary operation. Continue recessed filter development work and support for the on-line laser perforator for product development.

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PROJECT NUMBER: 1333
PROJECT TITLE: Semiworks Process Control
PROJECT LEADER: D. A. Phan
PERIOD COVERED: April, 1988

A. **Objective**: Evaluate and revise the process control and data acquisition system to improve processing performance and production quality.

B. **Results**:

ADT Dryer Control Improvements (Sims) - The design of a special data logging and retrieval system was complete. This system provides more accurate process data during the steady-state portion of all production runs through the Adt dryer for process analysis. The process report, generated from this system, includes averages and standard deviations of all key variables from the cutter steam cylinder to the final silos.

Second Bright Casing Flavor System (Oliver) - Electrical and instrumentation installation for a second bright casing flavor system was complete and checked out. This system will be on-line the week of May 9, 1988.

Hauni HT Steam Tunnel Installation (Oliver) - Work is underway to rearrange the Adt dryer infeed to accomodate the addition of a HT steam tunnel that is scheduled to be installed in July 88. This includes the modifications to the inlet feeder and weighbelt, and the installation of a new Corra-Trough conveyor after the cutters. This work is being done in conjunction with F. Boothe of R&D Development Engineering.

3,000-b Steam Cylinder Upgrade (Oliver/Medek) - Plan is in progress to upgrade the steam piping system at the 3,000-lb steam cylinder due to severe corrosion of steam piping at this location. The scope of work will also include the rearrangement of existing steam and water piping stand, and overall cleanup of unused valves and instruments.

Temperature Control System For Scandia Overwrappers (Phan/Sims) - Development work by J. Osmalov indicated that temperature setting of the end seal heaters is critical for pack seal quality, and the existing system provides poor temperature control (+/- 10 degrees F). Plan is underway to provide better temperature control system (to 2 degree F) for all end seal heaters.

C. **Plans**: Complete all software modifications on the primary backup computer. Complete the design of a new steam and water piping stand and order required parts for the 3,000 lb steam cylinder. Order instruments to improve temperature control of all Scandia overwrappers. Continue to provide electrical plant engineering support to the Semiworks and conduct routine QA function.

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PROJECT NUMBER: 1503
PROJECT TITLE: Modified Smoking Materials
PROJECT LEADER: J. G. Nepomuceno
PERIOD COVERED: April, 1988

I. FOAMED FILLER BINDER

A. **Objective**: Develop a process for applying a subjectively acceptable binder to the tobacco filler during making to improve cigarette quality.

B. **Results**: Test results using a binder formulation consisting of 48% maltodextrin and 5% licorice (added as a foaming agent) showed cigarette quality improvements in terms of firmness and loose ends reduction in preliminary testing conducted to screen the maltodextrin-based formulations. Based on these results, an experimental design utilizing extended production runs with this foamed binder solution is in progress at the Semiworks. These cigarettes are being produced with 0.8% binder solids added to the rod. In addition, an OV adjustment of 0.5% is being made to the test filler in order to compensate for the OV increase due to the binder addition. The first of these extended runs has been completed and the cigarettes are currently being tested for physical quality and subjectives. Flavor Development panel smoking results to date have shown significant differences between the control cigarettes (no binder) and the test cigarettes (0.8% binder addition), with negative attributes being associated with the test cigarettes. In this light, additional extended runs with the maltodextrin formulation have been postponed until maltodextrin from alternate suppliers (different processing conditions) can be screened subjectively. Hand-injected models have been prepared for subjective screening and will be smoked next week. Additional runs on the experimental design will be completed with the most preferred candidate.

C. **Plans**: Perform subjective screening of the maltodextrin from alternate suppliers and complete the experimental design with the most preferred candidate.

II. LOW DENSITY ROD (BINDER APPLICATION)

A. **Objective**: Produce pre-coated tobacco via the batch process to support the current product and process development efforts for the low density rod program. Develop a method of pre-applying binders to the tobacco in a manner that can be easily scaled up for commercial use.

B. **Results**: Initial trials with the continuous coating reel using a 4% pectin solution look promising. Modifications to the nozzle arrangement (to keep tobacco from accumulating at the nozzle) as well as modifications to the tobacco feeding system are still in progress. However, the initial trials showed no major problems in terms of applying a 3% coating of pectin on the tobacco. These trials with pectin were performed over a 30 to 60-minute period.

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PROJECT NUMBER: 1333
PROJECT TITLE: Semiworks Process Control
PROJECT LEADER: D. A. Phan
PERIOD COVERED: April, 1988

A. **Objective**: Evaluate and revise the process control and data acquisition system to improve processing performance and production quality.

B. **Results**:

ADT Dryer Control Improvements (Sims) - The design of a special data logging and retrieval system was complete. This system provides more accurate process data during the steady-state portion of all production runs through the Adt dryer for process analysis. The process report, generated from this system, includes averages and standard deviations of all key variables from the cutter steam cylinder to the final silos.

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Temperature Control System For Scandia Overwrappers (Phan/Sims) - Development work by J. Osmalov indicated that temperature setting of the end seal heaters is critical for pack seal quality, and the existing system provides poor temperature control (+/- 10 degrees F). Plan is underway to provide better temperature control system (to 2 degree F) for all end seal heaters.

C. **Plans**: Complete all software modifications on the primary backup computer. Complete the design of a new steam and water piping stand and order required parts for the 3,000 lb steam cylinder. Order instruments to improve temperature control of all Scandia overwrappers. Continue to provide electrical plant engineering support to the Semiworks and conduct routine QA function.

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PROJECT NUMBER: 1806
PROJECT TITLE: New Tobacco Processes
SECTION LEADER: S. R. Wagoner
PERIOD COVERED: April, 1988

I. PROJECT ART - PILOT PLANT SUPPORT

A. **Objective**: To provide processes for converting and casing stem materials for the Bermuda Hundred Pilot Plant.

B. **Results**: Batches of Louisville rolled/cut stems and shredded stems were cased with monopotassium citrate to various citrate and moisture levels and shipped to the Bermuda Hundred Pilot Plant.

C. **Plans**: Continue to produce stem products as required by Project ART. Complete the installation of the CRS line in D Pilot Plant so that variables such as cut width can be tested at BHPP.

II. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

A. **Objective**: To conduct trials providing information for development of the ART commercial process.

B. **Results**: Baseline evaluation of the Dickinson Admoist conditioner was completed by determining the conditioning potential of the unit as measured by OV and temperature gains. Statistical testing, at the typical Pilot Plant stem throughput of 1000 lb/hr, identified the impact of each operating variable: steam pressure to the sparge tube, steam pressure to the water nozzles, water flow to the nozzles, and residence time. Analysis of the data has shown that the exit stem temperature is primarily a function of the steam pressure to the sparge tube, while OV gain is directly related to the water flow rate and residence time. A production rate (~6000 lb/hr) simulation test was successfully conducted, conditioning whole stems from 28 to 31.5% OV with an average exit stem temperature of 195°F. A mechanical loading test was also run by lowering the angle of the Admoist such that the unit retained approximately the weight (500 lb) of stems that it will in production. This test was run for four hours and no significant processing problems were encountered.

The Admoist conditioner and the Quester steam tunnel were evaluated for post-ART filler treatment, i.e., elimination of clumps and effect on subjective response. For the Admoist trial, no judgement could be made on the unit's ability to declump filler, since the extraction run failed to produce clumps in the feed material. The treated filler will be chemically and subjectively evaluated to determine the effect of conditioning on the ammonia level. The Quester tunnel was unsuccessful in eliminating clumps in the post-ART filler. This filler will also be evaluated for subjective and chemical effects.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

Subjective testing of the post-ART stems (CRS) that were processed into superheated IS in Louisville indicated a tendency toward reduced liking scores when compared to the control IS at 5% levels in a Marlboro configuration. Initial comparisons in PM Superlights showed similar results. Further testing is in progress to confirm these findings.

C. **Plans**: Conduct trials to determine the optimum method (pneumatic, screening) of separating clumps from post-ART filler, such that, the clumps can be treated as a sidestream process.

Conduct subjective evaluations of post-ART stems at lower potassium citrate levels and using alternate casings, such as citric acid.

III. BINDER DEVELOPMENT

A. **Objective**: Develop methods to produce binder systems for the foam bound rod, bonded ends, and low density rod programs.

B. **Results**: Preliminary subjective testing of cigarettes using a NaCMC/licorice binder system has been positive in Engineering's bonded ends program. A meeting was held with a representative of Aqualon to discuss various forms of CMC. Samples of Ca, Mg, and K CMC will be received for comparison testing versus NaCMC.

Meetings were held with A. Henriksen of PM Australia and A. Schwarb of FTR to discuss the foam bound rod program. Test results and QA specifications for pectins and maltodextrins were presented.

Propyl paraben results from the second preapplied binder tracer trial were received from General Analytical. Again, the data showed approximately half of the paraben was found on the tobacco. In addition, only half of the paraben was detected on the samples that were hand sprayed with a propyl paraben spiked pectin solution. Plans include using fucose as a tracer.

C. **Plans**: Continue to provide support as required to the above programs.

IV. TMCI-ASTA SHEET

A. **Objective**: To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TMCI process and PM-RCB technology for international application.

B. **Results**: The Tarragona ASTA trials are scheduled to begin on May 9th and will take approximately three weeks to complete. Laboratory evaluation of more finely ground dust (<400 mesh) than specified by TMCI (<60 mesh) demonstrated improved sheet quality. The slurry viscosity of this finer dust with PM binder was reduced which improved casting uniformity. The sheet had a translucent appearance which indicates improved pectin release and it

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contained very few pinholes which should improve filler survivability through primary processing. Some differences in humectant analysis between PM and USA were identified and a PM analyst was sent to TSA laboratories in Madrid to resolve the differences before the trials begin in Tarragona.

C. **Plans**:

1. Complete the series of ASTA trials including evaluation of finer tobacco particle size in May.

2. Identify and evaluate the best ASTA product candidates for physical and subjective characteristics in USA by the end of August.

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PROJECT NUMBER: 1810
PROJECT TITLE: Project ART
PROJECT LEADER: Ravi Prasad
PERIOD COVERED: April, 1988

PROJECT ART

A. **Objective**: To support Commercial Plant Design and Flavor Development Objectives at the Bermuda Hundred Pilot Plant.

B. **Results**: Process operating parameters for nicotine extraction from DL blend, were optimized to give the target extraction level of 97% nicotine reduction with favorable subjectives. Filler AB application level of 2.5% at an extraction temperature of 140°F gave the best subjectives.

The stem absorber optimization work was initiated. Potassium citrate level on CRS stems was successfully reduced (from 17% to 12% K-citrate level) with same extraction efficiency of 97% nicotine removal.

An experimental grid to reduce the pressure drop in CO_2 extraction loop was initiated. Directionally, pressure drop can be reduced utilizing screens with greater open area.

Installation and startup of the on-line nicotine monitor was successfully completed. Initial data provided by the HPLC has been encouraging. Due to an immediate need for an "on-line" device for the commercial plant, this part of the R&D activity is being expedited.

A series of runs to document the advantages of keeping the absorber at a lower temperature than the extractor, was completed. A heat exchanger is being designed to allow "cooling" of CO_2 after the extractor to maintain the absorber at a lower temperature throughout the extraction cycle.

C. **Plans**: To continue support of Commercial Plant and Product Development objectives.

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PROJECT NUMBER: 1101
PROJECT TITLE: Entomological Research
PROJECT LEADER: D. L. Faustini
PERIOD COVERED: April, 1988

I. CIGARETTE BEETLE (CB) PHYSIOLOGICAL STUDIES

A. Objective: To conduct research investigations designed to produce results that lead to the control of the CB growth and development.

B. Results: The 5-day modified atmosphere tests using a nitrogen environment did not always demonstrate 100% CB mortality (1). Consequently, a 6-day exposure period was conducted. To date using 32,000 insects, 100% mortality has been observed.

Methoprene free tobaccos were blended and cut into filler. The filler was treated at 2.5 ppm (S-methoprene) and 5.0 ppm (R,S-methoprene) (2).

Iron oxide (oxygen absorber) sachets were evaluated as a control method for the CB in bags of cut filler. Polyethylene (PE) bags were dosed at 1, 2, or 3 sachets/PE bag. Preliminary results showed an average of 99, 92, and 100% CB control in the respective treatments. Survivorship was attributed to oxygen entering the PE bag through small holes created from the heat seal bar (3).

A carbon dioxide disinfestation was conducted at Park 500 Leaf warehouse #2 for a 9-day exposure at 30 ± 17% CO_2. This work was performed in collaboration with Leaf, E&EP, and QA. One hundred percent CB mortality was achieved for eggs, larvae and adults and 95% for pupae (4).

C. Plans: To obtain data necessary for ARS/APHIS approval of a modified atmosphere as a quarantine treatment, methoprene treated fillers will be made into cigarettes, packaged and shipped to Japan's Danchi Evaluation Panel. Iron oxide sachets will be evaluated against 40-100,000 CBs to gain ARS/APHIS approval as a treatment for export cut filler. The large scale disinfestation of tobacco warehouses using CO_2 will be recommended to the Pest Management Committee as an alternative to phosphine fumigations.

D. References

1. S. Drew. Memo to D. L. Faustini. Status and Results of Isolcell® Test for ARS. April 7, 1988.

2. R. M. Lehman. Notebook No. 8519, pp. 65-66.

3. R. M. Lehman. Notebook No. 8519, pp. 67-69.

4. D. T. Wagner. Memo to L. Ryan. CO_2 Fumigation Park 500. March 30, 1988.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

II. SERVICE TO OTHERS

A. **Objective**: To conduct and provide technical services to areas outside R&D.

B. **Results**: Two herbicides were added to the pesticide list (1).

Leaf Department was consulted regarding entomological experiments to be conducted in warehouse storages in 1988-89 (2).

Zoecon Corp. requested PM support for the use of methoprene by meeting with B.A.T. in May to discuss toxicology/safety of this material (3).

Assistance was provided to: the Turkish government regarding the subject of methoprene registration (4); TACASA (Guatemala) regarding the use of synthetic pyrethroids; PM Australia on the use of space sprays, pheromone traps and warehouse fumigations.

C. **References**

1. L. Ryan. Memo to T. Newman. Herbicides for Broadleaf Weed Control. March 30, 1988.

2. M. Minor. Memo to D. L. Faustini. Consultation with Leaf Dept. Management on Proposed Studies for 1988-89. April 15, 1988.

3. D. L. Faustini. Memo to R. W. McCuen. Meeting Scheduled with B.A.T. to Discuss the Safety of Methoprene. April 20, 1988.

4. D. L. Faustini. Letter to Dr. Gürbuz Capan. RE: Registration of Methoprene (Kabat® and Dianex®) in Turkey. April 20, 1988.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

PROJECT NUMBER: 1620
PROJECT TITLE: Electrophysiological Studies
PROJECT LEADER: F. P. Gullotta
WRITTEN BY: C. S. Hayes
PERIOD COVERED: April, 1988

I. NASAL EVENT-RELATED POTENTIALS (NERPs)

A. Objective: To develop methods by which to objectively and reliably evaluate human responses to cigarettes, smoke constituents and tobacco flavorants.

B. Results:

1. Cognitive NERP Study

Problems continue to be encountered with the new odorant delivery system of the olfactometer delaying the onset of cognitive NERP experiments. The problems appear to be related to the new birhinal switching device. Upon stimulation, (i.e., switching of the airstreams), a pulse (i.e., change in airflow) is detectable in both of the olfactometer airstreams. There must be no detectable change in flow during the switching of airstreams in order to assure artifact-free event-related potential recordings. Additionally, odorant is being released into the airstream during the inter-stimulus-interval (ISI), further indicating a problem with the switching and/or plumbing of the olfactometer. When correctly operating, the olfactometer should release a controlled amount of odorant into a clean airstream and then rapidly be flushed out. To date, these problems are awaiting investigation by J. Washington. Until they are corrected, experiments employing the olfactometer cannot be conducted.

Optimal methods for quantifying cognitive NERPs continue to be explored. To date, it appears that RS1 is neither powerful nor fast enough to handle the extensive amount of data generated during a principal component analysis. The BMDP software appears promising, but it is also very slow and would require an overnight batching of the data to the Unix system. Other statistical/graphical software packages continue to be evaluated for their utility in conducting principal component analyses and other multivariate analyses.

C. Plans: Once the above problems with the olfactometer are corrected, plans are to conduct pilot experiments to: 1) test the operation of the new system and, 2) determine the optimal parameters for elicitiation of the late positivity of the NERP. Following the fulfillment of these objectives, cognitive discrimination experiments comparing natural and synthetic menthol will begin.

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PROJECT NUMBER: 1702
PROJECT TITLE: Optical Processing and Aerosol Research
PROJECT LEADER: K. A. Cox
PERIOD COVERED: April, 1988

I. INDIVIDUAL CIGARETTE INSPECTION (D. Lowitz)

A. Objective: Develop methods for the inspection of individual cigarettes on a making machine.

B. Results: A CIM 7401 rod monitoring head and ancillary apparatus were obtained to permit setting up a test unit that is independent of the AccuRay 7000 module. With the help of A. Burton, the electronic portions of the test table were mounted and made operational.

C. Plans: Complete the CIM 7401 test unit by fabricating a sampling system. Interface the unit's output to a computer in order to permit the calculation and evaluation of potential optical or electronic filter functions. Determine the resolution limits of the CIM 7401 and the need for improvements utilizing acousto-optic cells.

II. OPTICAL PACK INSPECTION, EXPERIMENTAL (C. Harward)

A. Objective: Evaluate the Global Holonetics SMART CAMERA (SC). Determine its effectiveness in discriminating between good and defective packs.

B. Results: A video frame grabber and an optical disk drive were installed in a PC to allow a video image library of both good and defective cigarette packs to be collected. Global Holonetics personnel have modified the system so that video images can be recalled from the library and sent to the SC. This allows the SC to be shown the same set of images repeatedly in order to monitor its performance as a function of time. Two sets of images of cigarette packs have been collected for use in the evaluation of the SC.

III. OPTICAL CORRELATOR DEVELOPMENT (C. Harward)

A. Objective: Construct and test an optical correlator for pack inspection.

B. Results: An optical system was set up to fabricate an optical correlation filter. A specially modified LCD TV was used as a spatial light modulator (SLM) to encode a video image onto a laser beam. The corresponding filter was then obtained by recording the interference pattern produced between the encoded beam and a reference beam on a glass photographic plate. When this filter was used in an optical correlator, a small intense spot was

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observed in the center of the output plane when the corresponding image was displayed on the SLM. The intensity was observed to be reduced when other images were displayed.

C. **Plans**: Improve the technique used to produce the correlation filter. Determine the relative sensitivity of the correlation output to defective images and to acceptable variations in good images.

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PROJECT NUMBER: 1704
PROJECT TITLE: Supercritical Fluid Processes
PROJECT LEADER: T. M. Howell
PERIOD COVERED: April, 1988

I. LOW NICOTINE

A. **Objective**: Develop alternate scrubbing methods for nicotine removal.

B. **Results**: The water column was attached to the one liter lab system, and modifications were made to improve water level control. Two electrical conductivity probes indicate water level at a range of 1 to 5 cm above the column packing. After complete installation, the column and all associated plumbing were successfully pressure tested hydrostatically to 7500 psig.

After performing several shakedown trials, an extraction of an Art blend sprayed with 2.4% AB yielded 97.2% removal of nicotine. Excess water and CO_2 were employed to insure an accurate assessment of the viability of the water adsorber concept.

Analysis of the effluent ranged in nicotine concentration from 6.24 mg/ml to undetectable, and the material balance on nicotine closed to within 0.9%. Water samples were forwarded to Chemical Research for further analysis.

C. **Plans**: A duplicate run is planned to insure repeatability. Further work is planned in order to determine optimum design parameters for the water column.

II. LOW NICOTINE

A. **Objective**: Obtain equilibrium partition coefficients for nicotine between SC-CO_2 and K-citrate sprayed stems at various temperatures.

B. **Results**: The micro extraction system was re-fabricated in D201 and pressure tested to 7500 psig. A study to determine the background level of nicotine over the stems at 50, 55, and 60°C was initiated. Preliminary evaluations show CRS stems sprayed with 17% K-citrate at 32% OV have background levels of 3.7, 4.9, and 5.3 ppm nicotine, respectively, at the above temperatures.

C. **Plans**: Plans are to determine with multiple samplings the effects of temperature on the partition coefficients of stems and on fillers which contain very low levels of nicotine.

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PROJECT NUMBER: 1708
PROJECT TITLE: Physical Chemistry and Process Monitoring
PROJECT LEADER: J. L. Banyasz
PERIOD COVERED: April, 1988

I. OPERATIONS SUPPORT (J. Crump, in collaboration with the Applied Technology Group)

A. Objective: Develop a rapid method of moisture measurement for the stemmeries.

B. Results: Preparations are underway for the installation of a Humidat instrument in the stemmery on a trial basis. Seven Oriental type tobaccos have been equilibrated and are currently being tested to establish the Humidat's potential for moisture in these leaves.

C. Plans: Testing will continue to establish the Humidat's potential for Oriental leaf as well as for applications on the auction floor.

II. OPERATIONS SUPPORT (T. Van Auken and D. Gilliam, in collaboration with the Applied Technology Group)

A. Objective: Develop a rapid method of pectin determination.

B. Results: Sufficient quantities of tobacco pectin and fibrous material have been prepared for BL slurry to serve as standards for IA measurements.

C. Plans: Experiments will be initiated with the IA to establish a standard curve for pectin in BL slurry.

III. OPERATIONS SUPPORT (P. Henderson, in collaboration with the Applied Technology Group)

A. Objective: Characterization of inks.

B. Results: A considerable database has now been established for incoming inks using viscosity measurements and tack as measured by inkometer. B&H Ochre 60-928A has been of particular interest. Measurements have shown that within a batch this material is variable with relative standard deviations on the order of 15% for the viscosity and tack. Batch-to-batch variations are excessive, with the viscosity changing by as much as a factor of 2.5.

C. Plans: Preparations are being made to determine whether the vendor can provide a consistent product from batch to batch.

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IV. OPERATIONS SUPPORT (J. Crump and A. Closter, in collaboration with the Applied Technology Group)

A. Objective: Determine the effect of particle size distribution on dynamic viscosity changes that occur in PVA tipping adhesives.

B. Results: Measurements are underway in the simulator to determine the effect of machining on a series of base polymers of varying particle size distributions.

C. Plans: Tests on this series should be completed next month.

V. OPERATIONS SUPPORT (T. Van Auken, in collaboration with the Applied Technology Group)

A. Objective: Develop a rapid IA method for determining triacetin in filter plugs.

B. Results: A new sample holder for the IA to contain filter plugs has been designed and is under fabrication.

C. Plans: Once the holder is completed, experiments will be initiated to establish a calibration curve for triacetin in filter plugs.

VI. LOW DENSITY RODS (S. Ganeriwala and S. Shelton)

A. Objective: Compare the compression properties of individual low density and control rods.

B. Results: Sufficient data is now in hand to compare the dextran and pectin bound rods. Though both binders appear to bond tobacco shreds adequately, pectin stiffens tobacco significantly while dextran has little effect on stiffness. Rod compression tests show that dextran bound rods do not exhibit the initial sharp rise in rod firmness or stiffness characteristic of pectin bound rods. The compression curves for the dextran bound rods fall closer to the curves for pectin sprayed nonbonded rods than to those for the pectin bound rod. The results are consistent with the conclusion that increased shred stiffness plays a significant role in the firmness increase shown by bonded rods.

C. Plans: Work is underway to further develop the peel test so as to obtain a more quantitative comparison of bonding efficiencies.

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PROJECT NUMBER: 1720
PROJECT TITLE: Analytical Microscopy
PROJECT LEADER: V. L. Baliga
PERIOD COVERED: April, 1988

I. TIPPING PAPERS: CHEMICAL AND STRUCTURAL CHARACTERIZATION (Sanders)

A. **Objective**: Characterize chemical and structural morphology of tipping papers made with different concentrations of $CaCO_3$ and TiO_2.

B. **Results**: A request was made by B. Geiszler to examine cigarette tipping papers made by three different companies. Two of the papers, 12-A and 12-B, were made with 20-25% $CaCO_3$, 6-9% TiO_2, and 0-1% Al_2SiO_5. The other paper, 13-A, was made with the same amounts of Al_2SiO_5 but 5-10% $CaCO_3$ and 20-25% TiO_2. Each paper also had ~0.5g/m^2 nitrocellulose lip release coating and ink. The inner and outer surfaces of each paper were examined by high resolution, scanning electron microscopy.

There were differences in the size and shape of the crystalline additives used in the tipping paper.

C. **Plans**: Determine the elemental composition by EDS of each crystalline shape of the chemical additives.

D. **References**:

Sanders, K., PM Notebook #8612, pp. 25-34.

II. SAFETY (Sanders)

A. **Objective**: Instruct ERT members on basic human anatomy.

B. **Results**: An ERT first aid class on basic human anatomy was presented to the ERT members.

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PROJECT NUMBER: 1730
PROJECT TITLE: Plant, Cell & Tissue Culture Research
PROJECT LEADER: I. L. Uydess
PERIOD COVERED: April, 1988

I. TOBACCO IDENTICAL PRESERVATIVES

A. Objective: To develop procedures and to establish microbiological screens for the evaluation of new, nature-identical preservatives as replacements for and/or as adjuncts to propylparaben.

B. Status and Results:

1. Phase I, Preservative Screens:

a. Agar-Inclusion Assay:

Seven fatty acids ranging in chain length from C-6 to C-14 were tested in the scaled-down agar inclusion assay. The antimicrobial activity of these compounds was found to increase with increasing chain length up to and including Decanoic (C-10) and Undecylenic (C-11) acids, both of which completely inhibited microbial growth at 100 µg/ml. This is considerably better than the results achieved with propylparaben which inhibits bacterial growth at 300 - 500 µg/ml in this same assay system. The C-14 compound that was tested, Myristic (Tetradecanoic) Acid, did not inhibit bacterial growth completely until 5000 µg/ml.

b. Shake Flask Assay:

Two naturally-occurring compounds, a C-9 fatty acid (Nonanoic Acid) and a tobacco-identical sucrose ester extracted from Oriental tobacco were evaluated for antimicrobial activity in the Phase I (Nutrient Broth) shake flask assay. One hundred and fifty µg/ml of propylparaben in PG was used as the control. One hundred µg/ml of Nonanoic Acid reduced the growth of the target organism (B. coagulans) 70% compared to that of the preservative-free control. The control level of propylparaben (150 µg/ml) commonly used in these same experiments reduced the growth of the target organism 40% - 50%. Two hundred and fifty µg/ml of nonanoic acid totally inhibited the growth of the B. coagulans. The sucrose ester fraction, on the other hand, was observed to be even more effective as an antimicrobial agent since it completely inhibited growth at 100 µg/ml.

2. Microbiological Investigations:

a. Phase II screen: Passage of the mixed culture of bacteria isolated from laboratory-spoiled Park 500 SEL continued. Variations in apparent growth rate, final pH and relative proportion of each of the (4 - 5) different colony types were observed to occur during the spoilage cycle in each

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passage. These variations in population dynamics and chemistry of the spoiling SEL are thought to be the result of subtle and as yet, ill-understood metabolic interdependencies which exist among the organisms within the mixed culture. Each of the colony morphologies present in the mixed culture have been reisolated in an attempt to establish a single-isolate, SEL (Phase II) preservative screen, as well as to further investigate each isolate's contribution to the observations that have been made.

b. Spoilage of Starch-Based Adhesives: Nonanoic acid was incorporated into samples of a starch-based adhesive (National Starch and Chemical Corp.) and its antimicrobial activity evaluated. Duplicate aliquots of this material containing 0 to 1000 µg/ml nonanoic acid were dispensed asceptically into sterile petri dishes containing 10 mls of starch-based adhesive and exposed to the air in the Manufacturing Center glue room for 2 hours. The samples were then sealed, returned to the laboratory and incubated overnight at 21°C. Trypticase Soy Agar plates (+/- nonanoic acid) exposed to the air in the glue room and unexposed, adhesive-containing plates (sealed in the laminar flow hood) were included as additional controls. After 24 hours of incubation, a variety of mold and bacteria were observed on the control TSA plates (- nonanoic acid). Mold, but no bacteria were found on the TSA plates containing nonanoic acid at concentrations of 500 µg/ml and above. Mold was also observed on all of the starch-based adhesive plates, even those not exposed to the air in the glue room. Thus, there was a mold contaminant in the starch-based adhesive as supplied by the manufacturer that was not sensitive to the concentrations of nonanoic acid that were used. In a previous experiment (see March monthly), propylparaben was found to be effective against the mold present in those experiments.

C. Conclusions: None to be reported at this time.

D. Plans (May, 1988):

1. Additional fatty acids with chain lengths between C-10 and C-14 will be tested in the Phase I preservative screens.

2. Further work will be done on the development of a reproducible, single-isolate Phase II (SEL) screen.

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PROJECT NUMBER: 1752
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke
PROJECT LEADER: J. O. Lephardt
PERIOD COVERED: April, 1988

I. TANDEM MASS SPECTROMETRY

A. **Objective**: To evaluate for acquisition commercially available tandem mass spectrometer systems.

B. **Results**: Discussions were held with the commercial vendors and arrangements were made to visit the facilities of three vendors to coduct performance tests on their equipment. Visits to two vendors have been completed, and the third visit is scheduled for next week.

C. **Conclusions**: Distinct differences were found in both the ease of use of the two systems examined thus far and the general operation of the two companies involved. These observations supported the assessments obtained from present users of each companies equipment as to the responsiveness and service records of each company.

D. **Plans**: Upon completing the third site visit, assessment of the three system's relative merits for PM applications will be made and preparation of a formal recomendation and purchase request initiated.

II. SMOKE ANALYSIS

A. **Objective**: Mainstream smoke analysis of Trim cigarettes.

B. **Results**: Test cigarettes, coded P2123-42, G8IS, with a Trim II blend and 35% $CaCO_3$ of small particle size in the paper were compared with the Kentucky reference cigarettes, 1R4F, by fractionation of the mainstream smoke. In the basic fractions, 22 bases, including some pyridines, pyrazines and most of the minor alkaloids, show small but significant differences between the two cigarettes. The intensities of these compounds, except methylpyrazine and 2,6 dimethylpyrazine, favor the reference cigarette. Among the acidic compounds, 18 components are different including many phenolic compounds, aromatic acids and fatty acids. The intensities of the acidic compounds show more parity in these two cigarettes. In the neutral fraction, significant differences were seen for toluene, indole, 3-methylindole and four megastigmatrienone isomers.

C. **Plans**: Continue to provide support as required.

D. **References**:

Hsu, F. & M. Buckner, "Mainstream Smoke of Project Trim Cigarettes," memo to R. Cox, April 8, 1988.

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A. **Objective**: Determination of MS and SS Acrolein by TDL Spectroscopy

B. **Results**: The mainstream acrolein deliveries have been determined for 100% single blend component cigarettes which were part of the training set for the North Complex Smoking Panel. On a per puff basis, 100 % RCB delivered the lowest level (6.5 micrograms/puff) while 100 % ET delivered the highest level (16.1 micrograms/puff). FTR reference cigarettes C20 and C50 delivered approximately 9 and 4.5 micrograms/puff respectively.

C. **Conclusions**: In MS, acrolein deliveries are not dramatically different relative to the type of tobacco smoked.

D. **Plans**: The effect of casings and glycerine free blends will be evaluated for MS acrolein. The SS inlet system will be set up and SS measurements for the 100 % single blend components cigarettes will be made. Comparisons with the 2,4 DNPH aldehyde procedure will also be made.

E. **References**:

PM Notebook #8617, pp. 1, 2, 9.

A. **Objective**: To compare the organic gas phase of smoke from Project Trim cigarettes to that from 1R4F control cigarettes.

B. **Results**: No difference was seen in the OGPP from the Trim cigarettes when compared to the 1R4F controls.

C. **Conclusions**: The experimental conditions and results were described in a memo.

D. **References**:

Magin, D. F., "OGPP Analysis of Project TRIM Cigarettes," memo to R. Cox, April 5, 1988.

III. ASHTRAY ODOR

A. **Objective**: Fractionation of neutral fraction from ashtray odor.

B. **Results**: An absorption column packed with 40 um silica gel was used to further fractionate the neutral compounds isolated from cigarette butts. By eluting successively with hexane, methylene chloride, methylene chloride/methanol (9:1 by volume), and methanol, four fractions were obtained. By comparing the GC profiles and the resulting odor from each fraction, the fractionation was fairly reproducible. Fractions 1,3 and 4 had strong and distinct odors. All four fractions are being evaluated by the Odor Profiling Panel. The compounds in fractions 3 and 4 are being identified by GC/MS analysis.

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C. **Plans**: The odor intensity of those compounds in fractions 3 and 4 will be evaluated by GC/sniffing/MS. Their retention indices will also be measured and entered into a RS/1 table.

IV. PYROLYSIS-GC/MS

A. **Objective**: To examine CR- Compounds using the technique of Curie Point Pyrolysis-GC/MS.

B. **Results**:

Samples run during the month of April included: CR-2704, -2706, -2722, -2513, -2514 (for J. Paine); CR-2719 (for R. Izac); CR-2643 (for K. Podraza). Each of these was pyrolyzed at two temperatures, the pyroyzates chromatographed using fused silica capillary column GC, and the peaks detected and identified using mass spectrometry.

In addition, the results of last month's pyrolysis-GC/MS of samples CR-2707, -2708, -2711, and -2712 (for R. Izac), were reexamined in order to determine the ratio of menthol to menthenes and the relative release of menthol upon pyrolysis of these compounds.

C. **Conclusions**: The results of these experiments were conveyed to the originators of the samples in memos. The reexamination of the results of the samples from last month was communicated to R. Izac.

D. **References**:

1. Magin, D. F., "Pyrolysis-GC/MS of CR-2719," memo to R. Izac, April 8, 1988.
2. Magin, D. F., "Pyrolysis-GC/MS of CR- Compounds," memo to J. Paine, April 11, 1988.
3. Magin, D. F., "Pyrolysis-GC/MS of CR-2643," memo to K. Podraza, April 20, 1988.

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Source: https://www.industrydocuments.ucsf.edu/docs/rffm0000

PROJECT NUMBER: 1754
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components
PROJECT LEADER: J. B. Wooten
PERIOD COVERED: April, 1988

I. SOLUTION NMR OF TOBACCO COMPONENTS

A. **Objective**: Determine the composition and structure of tobacco cell wall biopolymers (J. Wooten, S. Baldwin).

B. **Results**: The ^{13}C NMR spectrum of tobacco hemicellulose was obtained and the results were correlated with the the neutral sugar analysis. The sample was found to contain 40% glucose, 17% galactose, 16% xylose with smaller amounts of other sugars. Not surprisingly, the NMR spectrum of such a complex material is complicated, but numerous distinct spectral features were observed including 7 resolved anomeric carbon resonances. Of particular note was the absence of any signal at 103.2 ppm. The anomeric carbon signal of glucose having two contiguous glucose residues appears at 103.2 ppm. This indicates that the hemicellulose does not contain significant Glu-Glu-Glu units (or larger Glu groups) and that the glucose is distributed randomly with the remaining sugars. The analysis of this material is continuing.

A. **Objective**: NMR analysis of 2-cyclohexene-1-one derivatives (R. Bassfield, K. Podraza).

B. **Results**: The complete spectral assignments of the ^{1}H and ^{13}C NMR spectra of five cyclohexenone derivatives were made using one and two dimensional NMR techniques. The ^{1}H-^{1}H coupling constants were used to define the orientation of the alkyl substituents on the cyclohexenone ring.

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PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: C. S. Kroustalis
PERIOD COVERED: April, 1988

ANALYTICAL FLAVOR SPECIFICATIONS

A. Objective: Develop analytical and sensory specifications for incoming flavor materials for use at the Flavor Center and other QA facilities.

B. Results: Methods for sample preparation and analysis were written to be included with the final specifications. We are currently reviewing the draft specifications and we are including the method used for the evaluation of each material. Uniform methodology for the evaluation of bulk materials is addressed in collaboration with Mr. D. Watson.

C. Plans: Issue final specifications for the materials received to-date to Mr. F. Daylor. Begin the evaluation of domestic materials on a vendor by vendor basis at a rate of 15 to 20 per month.

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PROJECT NUMBER: 1758
PROJECT TITLE: Tobacco Cell Wall Research
PROJECT LEADER: G. H. Bokelman
PERIOD COVERED: April, 1988

I. ESTIMATION OF BLEND COMPOSITION FOR CIGARETTES SOLD IN JAPAN

(G. Bokelman, J. Stimler and General Analytical)

A. Objective: Predict the blend compositions of Salem Lights (Japan), produced by RJR Nabisco, and Viceroy Milds (Japan), produced by B&W. Both cigarettes are manufactured in the U.S. for sale in Japan.

B. Results: Since Islands and Salem Lights (Japan) are both produced by RJR Nabisco, we were interested in comparing these brands. It previously was reported (1) that, compared to the five brands produced by JTI which we have examined, Islands is notable for its low content of bright lamina and its extremely high content of Oriental leaf. The predicted blend compositions of Libera Milds, produced by JTI, Salem Lights (U.S.), Viceroy Milds (Japan) and Viceroy Kings (U.S.) also have been included in the table below (2).

Predicted Blend Compositions
(values expressed as percentage of total)

Component	Salem Lights (U.S.)	Salem Lights (Japan)	Islands (RJR for Japan)	Libera Milds (JTI)	Viceroy Milds (Japan)	Viceroy Kings (U.S.)
Bright Lamina	27	36	35	41	31	39
Burley Lamina	36	24	18	33	32	27
Oriental Leaf	19	16	24	8	10	12
Stem	19	24	23	18	28	22

Compared to Salem Lights (U.S.), Salem Lights (Japan) has more bright lamina and (combined bright and burley) stems, but less Oriental leaf and much less burley lamina. These are all "reasonable" changes to make considering the different trends in blend composition for the two markets. Compared to Islands, Salem Lights (Japan) has more burley lamina, less Oriental leaf, and about the same contents of bright lamina and (combined bright and burley) stems.

B&W made some interesting changes in Viceroy Milds (Japan) compared to its domestic brand of Viceroy Kings. The content of burley lamina was increased, but the bright lamina content was dropped significantly. The content of Oriental leaf was held about the same; however, the quantity of (combined bright and burley) stems was significantly increased. Libera Milds and Viceroy Milds have more burley lamina than any other brands on the Japanese market that we have examined. After them comes Marlboro Lights (Japan).

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C. **Plans**: A series of cigarettes produced in Thailand have been submitted for chemical analyses. As soon as these analytical values are available, we will estimate their blend compositions.

D. **References**:

1. Bokelman, G. H. and J. O. Stimler, memo to C. L. Irving, "Blend Composition of Islands," March 17, 1988.

2. Bokelman, G. H. and J. O. Stimler, memo to C. L. Irving, "Blend Composition of Salem Lights (Japan) and Viceroy Milds (Japan)," April 20, 1988.

II. TOBACCO HEMICELLULOSE (S. Baldwin and N. Lewis)

A. **Objective**: Characterize hemicellulose isolated from green Coker 319 tobacco.

B. **Results**: As part of a collaborative contract research project, Dr. Norman Lewis at VPI&SU sent us a crude sample of hemicellulose that had been extracted from green Coker 319 tobacco grown in the greenhouse at Philip Morris. It was determined that the carbohydrate content of this sample is 85-90%. A neutral sugar analysis that was performed on this material yielded the following results: rhamnose, 3.1%; fucose, ~0%; arabinose, 5.9%; xylose, 15.7%; mannose, 5.3%; galactose, 16.8%; and glucose, 40.4%.

Not all of the crude starting sample could be dissolved in water. In addition to the insoluble material, gel permeation chromatography revealed the presence of no fewer than three components. It is believed that at least one of these components is a xyloglucan polysaccharide.

C. **Plans**: The individual components within this sample of tobacco hemicellulose will be isolated and more completely characterized.

III. APPLICATION OF CMC TO CIGARETTE WRAPPER (S. Baldwin and B. Goodman)

A. **Objective**: Examine the effect upon sidestream of applying sodium carboxymethylcellulose (CMC) to cigarette wrapper.

B. **Results**: A low viscosity CMC was applied to cigarette paper which had a basis weight of 25 g/m^2. The CMC was applied in the Semiworks by size press application at levels of approximately 0.5%, 1.0%, 1.5% and 2.0%.

C. **Plans**: Cigarettes will be prepared from these treated papers in order to evaluate reduction in sidestream smoke.

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PROJECT NUMBER: 1902
PROJECT TITLE: Tobacco Microbiology
PROJECT LEADER: D. J. Ayers
WRITTEN BY: J. B. Jones
PERIOD COVERED: April, 1988

I. SHREDDED STEM STUDY (FOR PROJECT ART)

A. **Objective**: To determine if the ART process results in changes to the microbial numbers in shredded bright stems (12% and 35% OV's) after being stored for 0, 24, 48, 72, and 96 hours and 1,2,4,8, and 12 weeks at 37°C (3).

B. **Results**: Shredded bright stem samples (12% and 35% OV) were tested before the citrate spray (12% OV), after the citrate spray (35% OV), and post-ART dried or "as is" (12% and 35% OV, respectively). Results from up to 12 weeks of storage showed no significant increase in bacterial counts (1,2). Mold and yeast showed a steady decline and remained within an acceptable range.

C. **Plans**: This is an ongoing study.

D. **References**:

1. Jones, J. Notebook No. 8590, pp. 6-20, 21, 23, 25, 27, 28.

2. Ayers, D. Monthly Summary Acc. No. 88-022, March, 1988.

II. SHREDDED STEM STUDY (MICROBIAL STABILITY OF POST-ART SAMPLES)

A. **Objective**: To determine if post-ART treated stems (35% OV) can be stored for up to 24 hours without significant change in microbial numbers.

B. **Results**: Although quite variable, bacterial counts indicated no statistically significant increase during the 24 hours of storage. Mold and yeast were below 10 CFU/g stem and did not increase during the study (1). Low molecular weight organic acids were run at hours 0 and 19 and indicated that no significant changes occurred in these parameters (2).

C. **Plans**: Document results.

D. **References:**

1. Gaines, O. Notebook No. 8505, pp. 144-145, 151-156.

2. Crockett, E. Notebook No. 8563, pp. 84-90, 122-134.

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III. SHREDDED STEM STUDY (STORAGE OF POST-ART DRIED SAMPLES)

A. Objective: To determine the effect of long-term storage on microbial counts in the post-ART dried samples (12% OV).

B. Results: After 4 weeks of storage the post-ART dried samples from experiments 1,4, and 5 exhibited a decrease in bacterial counts as compared to initial counts. Experiments 2 and 3 showed no change in bacterial counts. Mold and yeast counts were within an acceptable range (1).

C. Plans: Complete 8 and 12 week storage samples.

D. References:

1. Crockett, E. Notebook No. 8563, pp. 79-83, 91-115, 135.

IV. RL/RCB ALTERNATE HUMECTANT PROGRAM (PARK 500 AND BL PLANT RUNS)

A. Objective: To determine if RL and RCB, produced as part of the Alternate Humectant Program, differ in microbial numbers between control and test sheets (1).

B. Results: RL control and test sheets did not indicate a difference in bacterial, mold, or yeast counts with the time 0 (T=0) samples (2). RCB sheets showed slightly higher bacterial counts on the 2 samples with no class tobacco (special control and test) as compared to the control with class tobacco. Mold and yeast remained within an acceptable range for both RL and RCB samples.

C. Plans: This is an ongoing study for the RCB sheets.

D. References:

1. Mooz, E. Second Revised Plan for the Alternate Humectant Program for 1988 : Production of a PG/G-Free Cigarette, 20 January 1988.

2. Jones, J. Notebook No. 8590, pp. 26, 29-31, 34-35.

V. HOGSHEAD, BOX, AND BALE STUDY FOR BURLEY (1987 CROP YEAR)

A. Objective: To determine if differences exist in microbial numbers, over a 3 year period, in Burley stored in Hogsheads, Boxes, and Bales in the Commerce Road Warehouse.

B. Results: Bacterial counts show fluctuations within and among the different container types (1). There did not appear to be a statistical difference in bacterial counts among the 3 types of containers. The majority of samples decreased or remained the same in mold and yeast counts. A memo has been written for crop year 1986 (2).

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C. **Plans**: Crop Year 1987 samples will be done in April, 1988.

D. **References**:

1. Crockett, E. Notebook No. 8563, pp. 42-50, 80-94, 113-115.

2. Crockett, E. Microbial Analysis of Burley Tobacco as Part of the Hogshead, Box, and Bale Study (1986 Crop Year After 1 Year of Storage), 15 April 1988.

VI. VALIDATION OF THE BACTOMETER

A. **Objective**: To determine the effect of spores versus vegetative cells on detection time in the Bactometer.

B. **Results**: Two experiments have been completed to date and results are being analyzed (1).

C. **Plans**: Repeat the experiment.

D. **References**: Chadick,D. Notebook No. 8265, p. 27.

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PROJECT NUMBER: 1904
PROJECT TITLE: Tobacco Physiology and Biochemistry
PROJECT LEADER: H. Y. Nakatani
PERIOD COVERED: April, 1988

I. LOW NICOTINE STUDY

A. **Objective**: To investigate the biochemistry of the nicotine biosynthetic pathway at putrescine N-methyltransferase (PMT) and specifically to isolate PMT from tobacco root extracts.

B. **Status**: Extraction of the roots from the group ten plants were initiated to the 40-65 % ammonium sulfate stage; the pellets were resuspended in 200 ml of buffer plus 1.5 M NaCl (2).

The Sephacryl 200 (Pharmacia) column has been calibrated with molecular weight standards run in pairs and in one experiment with the complete complement of four standards (3,4). In the latter case, there was an anamolous elution volume for chymotrypsinogen which did not fall on the standard curve (4). The reason for this phenomenon is unknown. Various samples from the radial flow phenyl-Sepharose column have been applied to this column and the PMT activity was eluted with an estimated molecular weight of 56,000 +/- 6000 Da for 4 sets (3,4,5). A second Sephacryl column is being set-up in the laboratory with an LKB fraction collecting system (4,5). Both Sephacryl columns will be run using this system.

Recently the S-adenosyl-homocysteine affinity columns were not found to bind PMT, and a series of experiments were conducted with the various batches provided by Bethesda Research Lab. One type (lot no. 71101), having both free amino and carboxyl termini was found to bind PMT. The PMT was eluted with 1.5 M NaCl (1). Moreover, a reactive red (a triazine dye) affinity column was also found to bind PMT. The binding was rather weak, however, since most of the PMT was eluted by simple washing with dialysis buffer. The reactive red was assumed to bind to proteins requiring nicotinamide as cofactor (1).

Several attempts were made to elute PMT from DEAE-Sepharose with a pH gradient. The results were negative although 5 mM phosphate, pH 6.5 and 5mM citrate-phosphate, pH 4.0 were used to develop the gradient. In another experiment 5 mM phosphate, pH 6.5 was paired with 10 mM citric acid, pH 2.0. The actual environment associated with the binding of PMT to this support medium may not have been affected by the conditions applied.

Another PMT (HPMT2) active sample was delivered to Hazleton for boosting the antigen response of the inoculated mice. A Western blot examination of this sample with the mouse antibody from mouse #5 was conducted using the polyvinylidene difluoride (PVDF) membrane and the new miniblotting device (5). A positive response

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was observed whereas no response was observed with the control serum (5).

A MagniSort M bead experiment using two levels of MagniSort beads was conducted with the HPMT2 sample. A decrease of approximately 40 % in the PMT-activity with the lower bead level was observed. In addition, PMT activity was "released" from the beads following 1 M $MgCl_2$ washing (5). These results certainly show promise for the antibody program.

The miniblotter was demonstrated at Hazleton for the examination of the third test-bleed against a Western blot with samples in 10 lanes (5). The examination could not have been conducted using their classical antibody detection methods since 5 ml of antibody would have been required.

C. **Plans**: The roots from the group 11, hydroponically-grown, tobacco plants will be harvested. The purification of PMT will continue with various chromatographic media. Antibody binding studies will continue using MagniSort-beads, Western blots and microtiter plates.

D. **References**:

1. Malik, V. S. Notebook No. 8542.

2. Dunn, R. L. Notebook No. 7899.

3. Sykes, A. Notebook No. 8526.

4. Yu, T. Notebook No. 8381.

5. Mooz, E. D. Notebook No. 8599.

II. ALTERNATE HUMECTANTS (PG/G-FREE SHEETS/CIGARETTES) (1)

A. **Objective**: To find a plasticizer/humectant system that provides acceptable sheet materials without glycols (PG/G).

B. **Results**: PG/G-free RCB was made at the BL Plant in April (2). Samples of the PG/G-free RCB sheet and samples of the PG/G-free RL (RLTC and RL150B) sheets that were made at Park 500 in March were submitted to Microbiology and the Analytical Research Division for testing.

C. **Plans**: 100% cigarettes will be made from the PG/G-free RCB and RL sheets, along with the controls. Second quarter plans for the Alternate Humectant Program for 1988 will be issued by the end of April.

D. **References**:

1. Mooz, E. D. Notebook No. 8599.

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2. Mooz, Elizabeth D. Request for the BL Plant to Produce PG/G-free RCB in Support of the Alternate Humectant High Priority Program. Memo to Mr. W. G. Overstreet; 1988 April 14.

III. ALTERNATE HUMECTANTS (GLYCERINE-FREE SHEET/CIGARETTES) (1)

A. **Objective**: To provide acceptable glycerine-free (G-free) RLTC, RL150B, and RCB sheets and G-free cigarettes for the European market.

B. **Results**: Domestic, G-free Marlboro-type cigarettes were made from Park 500 and BL Plant G-free sheets, G-free ET and ESB, and G-free casings and flavors. The cigarettes, including a control, were given to Cigarette Testing and to Flavor Development for evaluation. A three-month survivability test was run in the Semi-works on G-free RL (RLTC and RL150B) sheet that was made at Park 500 in 9/87.

C. **Plans**: A POL test is scheduled to be sent out the end of April to compare a domestic, G-free Marlboro vs a domestic, control Marlboro.

D. **References**:

1. Mooz, E. D. Notebook No. 8599.

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PROJECT NUMBER: 2106
PROJECT TITLE: Cigarette Performance and Design
PROJECT LEADER: R. W. Dwyer
PERIOD COVERED: April, 1988

I. CIGARETTE DELIVERY MODELING (J. Kao and B. Dwyer)

A. Objective: Develop predictive equations for relating cigarette performance to design.

B. Results: Tennessee Eastman's filter research laboratory has provided us with an extensive list of cellulose-acetate cigarette filter properties. Their experimental program included filter pressure drops and smoke removal efficiencies for unusually broad ranges of fiber size, number of fibers, filter length and filter circumference. We are using these data to develop models of vapor and particulate filtration as functions of filter design.

C. Conclusions: A semi-empirical model of smoke filtration efficiency has been developed. This model surpasses other models currently in the literature in terms of accuracy. A theoretical approach is being pursued to partition the total efficiency into particulate and condensation contributions.

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PROJECT NUMBER: 2500
PROJECT TITLE: Fundamental Chemistry
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: April, 1988

I. **FLAVOR/ODOR CHEMISTRY** (Paine, Secor)

A. **Objective**: To develop new technologies for smoke deliveries of desired flavorants; to prepare new substances for flavor/odor evaluation; to develop methodologies for the analysis of subjective data; to derive relationships between physicochemical parameters and subjectives.

B. **Results and Plans**: A request for two gallons of glucose menthyl carbonate (GMC) was met. Approximately 9 liters of 65% GMC by weight in PG was prepared and delivered to Flavor Development.

High purity samples of two tetramethylpyrazine (TMP) release agents were prepared. These two substances are part of a series of TMP-polyalcohol substrates, designed to have low volatility but high yields in thermal decomposition to TMP. A group of TMP release agents were submitted to Don Magin for pyrolyis-GC/MS and in all cases TMP was formed.

II. **CHEMICAL PHYSICS STUDIES OF TOBACCO CONSTITUENTS** (Secor, Seeman)

A. **Objective**: To obtain structural information about important tobacco constituents/flavorants; to develop information on cluster formation and chemical reactions in clusters.

B. **Results and Plans**: Experimental results previously obtained with E. Bernstein and his group (Colorado State University) on styrenes and anethole derivatives and on pyridines and pyrazines were evaluated. Reports on styrenes, α-methylstyrenes, and allylbenzenes are near completion. The preparation of two deuterated substrates [1-ethoxy-4-ethyl-d_5-benzene and 2-ethyl-d_5-pyridine] was completed and sent to CSU. Efforts continue to establish and to examine chemical reactions in clusters.

III. **REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS** (Secor, Seeman)

A. **Objective**: To develop techniques to remove nicotine and other tobacco alkaloids from aqueous tobacco processing fluids to the exclusion of all other components.

B. **Results and Plans**: Together with H. Grubbs, safety information regarding experimentation with nicotine was transmitted to Sepracor. Samples of the aqueous scrubber from the ART process was prepared by J. Dobbs, extracted into neutral and basic components, and analyzed by J. Naworal (GC/MS).

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PROJECT NUMBER: 2501
PROJECT TITLE: Smoke Chemistry
PROJECT LEADER: R. Comes
PERIOD COVERED: April, 1988
WRITTEN BY: D. Douglas

I. SIDESTREAM SMOKE

A. Objective: Conduct analyses on mainstream and sidestream smoke to determine qualitative and quantitative differences between control and reduced visibility cigarettes. Develop methods to analyze for selected components. Demonstrate effective methods for reduction or removal of these components.

B. Status: (1) Quantitative amounts of specific compounds are being added to cigarettes. Their sidestream smoke is then analyzed using silica gel collection and elution techniques. The purposes of this work are to correlate subjective data with this analytical data and to examine the sensitivity of the analytical method to changes affecting the odor or other subjective qualities of the sidestream smoke. (2) Sidestream and mainstream smoke from a 17 mm circumference cigarette was compared to that from Ky. Ref. 1R4F cigarettes using the silica gel collection and fractionation procedure followed by gc analysis.

C. Conclusions: (1) Preliminary results indicate that analytical gc data obtained by this method produce obvious differences only when significant differences in odor are present. (2) No notable differences were found by this method.

D. Plans: Gas phase analysis techniques are being developed to compare sidestream smoke samples generated from conventional and various low sidestream cigarette models. Concentration-desorption techniques are a part of this effort. Additional work is in progress to define the ability of the silica gel technique to identify specific component differences in sidestream samples derived from different types of cigarettes.

II. MISCELLANEOUS

A. Objective: (1) Construct an environmental chamber to measure components of sidestream smoke under carefully controlled conditions. (2) Conduct sidestream smoke visibility measurements as required. (3) Upgrade computer and instrumental capabilities for the project. (4) Determine experimental conditions necessary to support the program to incorporate selected ingredients into cigarette models to improve sidestream smoke properties.

B. Results: (1) Modification of the lab/removal of old lab furniture, etc. will be initiated in May. Chamber design specifications have been sent to a vendor for preliminary plans and cost estimates. (2) Trim models have been completed. RL/humectant-study models are being measured. (3) The HP-MSD is now able to acquire data with one data station while different data is simultaneously analyzed with the other. A second

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plotter has been interfaced to the MSD. Procedures have been written to allow the mass spectral data for an entire chromatogram to be analyzed unattended. (4) The gc-ms analysis of vanillin (produced by pyrolysis of glucovanillin) in mainstream and sidestream smoke was facilitated by the use of cool, on-column gc injection.

C. **Conclusions**: (1) The environmental chamber project is currently concentrating on lab renovation and chamber design specifications. Preliminary planning of instrumentation and data handling aspects of the project has been conducted. This includes determining specifications for the types of microcomputers needed for data collection and reporting. Discussions with vendors of analytical equipment have emphasized concentration techniques, particulate measurement techniques and an investigation of the feasibility of FTIR as a technique for measurement of some components. (2) As expected, the sidestream smoke visibility is decreased with the reduced circumference cigarettes. Additional reductions occur with small-particle-size additives. (3) The changes in configuration and enhancements of data analysis have at least doubled productivity for this instrument and its operator. (4) Use of excessive heat in the analysis of vanillin will degrade the sample.

D. **Plans**: (1) Details of the finalized chamber design will be specified and final vendor selection will be made. Appropriate analytical instrumentation is being evaluated so that the chamber design will incorporate features necessary for the proper use of the instrumentation. (2) The smoke visibility apparatus will be moved to a conditioned room to maintain constant environmental conditions and to determine whether the temperature and humidity conditions in a conditioned environment affect visibility. (3) Continue gc-ms analyses for sidestream studies. Adapt the concentration procedure and/or valve-operated multi-dimensional chromatography to this instrument to effect additional compound identifications. (4) The vanillin analysis will be completed. Several Project Art samples will be analyzed by gc-ms.

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PROJECT NUMBER: 2507
PROJECT TITLE: Radiochemical Investigations
PROJECT LEADER: S. B. Hassam
PERIOD COVERED: April, 1988

SIDESTREAM SMOKE

A. **Objective**: Develop and apply methods for collection and radiochromatography of SS from cigarettes labeled with ^{14}C-glycerol.

B. **Results**: (1) Development of a method to measure radioactivity in SS gas phase from ^{14}C-glycerol cigarettes using the new gas chromatography radioactivity detector is in progress. Preliminary investigations show that the detector sensitivity will be adequate to measure the amount of activity present in SS; the lower activity present in MS gas phase requires larger sample volumes to be injected, with the potential problem of overloading the GC column. Currently a GS-Q megabore column is being used for separation of gas phase components. Chemical detection of peaks is done with a TCD positioned prior to the radioactivity detector. Problems have been experienced with the TCD when the column is operated below ambient temperature. Attempts to correct this by gas flow modulation resulted in loss of sensitivity and resolution.

In order to facilitate sample introduction and to provide a reproducible means of sample injection, a six-way Valco valve with a sample loop (250 µL) was installed on the gas chromatograph. The valve was connected to a calibration gas mixture cylinder via a tee, which also allows the introduction of ^{14}C-samples or other gases into the column.

(2) Using TLC and RP-HPLC, radiochromatography of samples from ^{14}C-glycerol-sprayed filler (code 10-21-87) was completed. An ethanol extract of the filler and a 2,4-DNPH extract of the filler showed all activity to be associated with ^{14}C-glycerol. This indicates no degradation of the radiolabeled filler occurred prior to smoking. Smoke samples from ^{14}C-glycerol cigarettes (see March monthly report) were concentrated at ambient temperature under a stream of nitrogen gas. Radiochromatography of these samples showed changes in the relative amounts of ^{14}C-glycerol vs. ^{14}C-products present in the original samples, presumably due to physical losses and/or chemical destruction. Nevertheless, only previously present ^{14}C-products were observed, namely acetaldehyde in SS traps and HCHO in the smoking chamber wash. There is either no ^{14}C-acrolein present, or the amount is at the level of background activity.

(3) Summaries of various aspects of the SS/glycerol project are being written.

C. **Plans**: Continue evaluation of the gas radiochraomatography system; continue documentation of completed work.

D. **References**: NB 8386, 8630, 8631 and 8610.

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PROJECT NUMBER: 2520
PROJECT TITLE: Flavor Research
PROJECT LEADER: E. W. Southwick
PERIOD COVERED: April, 1988

I. GLUCOSE-DERIVED FLAVORANTS

A. **Objective**: To develop glucose-derived flavorants.

B. **Results**: (1) Menthol-glucose carbonate. Reaction of glucose and 1-menthyloxycarbonylimidazolide in either pyridine or dimethylformamide in the presence of about 20 mol% of 1,8-diazabicyclo[5,4,0]undec-7-ene produced a reaction mixture which consisted of mainly 6-O-, 2-O- and β-1-O-menthyloxycarbonyl-D-glucoses, with the 6-O-isomer being the more abundant one. The 4th isomer was identified by C-13 NMR as the 3-O-menthyloxycarbonyl-D-glucose. The concentration of this isomer is relatively small compared with the others. However, its presence interfered with the chromatography such that a realistic estimation of the isomer distribution is not possible.

(2) Glycoside Synthesis. Reaction of phenethyl alcohol with N-iodosuccinamide and 3,4,6-tri-O-acetyl-D-glucal in acetonitrile gave a 55% yield of phenethyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-α-D-mannopyranoside. This reaction is designed as one of the model reactions leading to the total synthesis of the natural "sucrose ester".

Reaction of 2-ethoxy-5-(1-propenyl)phenol with 1,2,3,4,6-penta-O-acetyl-D-β-D-glucopyranose in benzene with a catalytic amount of boron trifluoride etherate gave a modest yield of the corresponding β-glucopyranoside. After removal of the blocking groups, the 2-ethoxy-5-(1-propenyl)phenyl β-D-glucopyranoside so obtained will be submitted as a candidate for mainstream/side-stream analysis.

(3) Spray-dried flavorants. A Yamato GB-22 Spray Dryer was received and put into operation. Trial runs using sodium chloride solutions were successful. Trial runs using glucose or sucrose solutions were far from satisfactory. The final products, being somewhat hygroscopic, tend to absorb some moisture and adhere strongly to the side of the vessel. Solutions of β-cyclodextrin were spray-dried successfully. The product also tends to adhere to the side of the vessel due to electrostatic charge. We are exploring various ways to minimize this phenomenon. In all the cases studied, the recovery is in the 60-70% range.

(4) Cyclodextrin complexes. Due to the recent cost reduction of β-cyclodextrin and a perceived need for a tetramethylpyrazine release agent, a complex of tetramethylpyrazine and β-cyclodextrin has been synthesized. A particular advantage to the use of the flavorant tetramethylpyrazine results from it having a high odor threshold and a low flavor threshold (Extra project, 0.5 ppm). The complex was synthesized by dissolving β-cyclodextrin in water

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followed by addition of tetramethylpyrazine. The mixture was stirred for 24 hours at room temperature. The precipitate was filtered, washed with 95% ethanol and ether. The solid material was dried under vacuum (0.01 mm Hg) for 4 hours. A 37% yield (8 grams) of the complex was obtained. Tube thermolysis indicated that tetramethylpyrazine was released upon decomposition of β-cyclodextrin (250-300°C). The amount of complexed tetramethylpyrazine was 5% on a weight basis as determined by thermolysis and GC analysis utilizing an external standard of tetramethylpyrazine.

II. PROJECT EXTRA

A. **Objective**: To develop proprietary flavor additives for enhanced flavor perception in low delivery cigarettes.

B. **Results**: (1) 6-Methylhexahydro-2(3H)-furanone (CR-2643). An investigation into the large scale synthesis of the precursor to CR-2643 was conducted. The precursor to CR-2643 is 6-(ethoxycarbonylmethyl)-3-methyl-2-cyclohexen-1-one (1). The reaction of 3-methyl-2-cyclohexen-1-one (0.1 mole scale) with lithium diisopropylamide was conducted in tetrahydrofuran at -78°C utilizing ethyl bromoacetate. This reaction yielded approximately 14 grams (80%) of 1. If the reaction was repeated utilizing ethyl chloroacetate instead of ethyl bromoacetate no alkylated product 1 was obtained. The material isolated contained mainly starting material. The effect of temperature on this reaction was investigated. If the reaction was conducted at 0°C utilizing ethyl bromoacetate only a 51% yield of 1 was obtained with a considerable amount of polymeric material being formed. Therefore, a good yield of 1 can be obtained on a large scale if ethyl bromoacetate is used and the reaction is conducted at -78°C.

Reduction of 6-(ethoxycarbonylmethyl)-3-methyl-2-cyclohexen-1-one (PtO_2, acetic acid) and cyclization of the resulting hydroxy ester (acetic acid, heat) resulted in the isolation of 6-methylhexahydro-2(3H)-benzofuranone. This reaction procedure is simple and most importantly generates the title compound with a higher ratio of the trans isomer (cis/trans ratio 45/55). A 2.5 g sample of 6-methylhexahydro-2(3H)-benzofuranone (cis/trans 45/55) is now available.

(2) Acylpyrazines and derivatives. Acylation of 2,5-dimethyl-pyrazine with acetaldehyde gave the desired 2-acetyl-3,6-dimethyl-pyrazine in good yield following purification by reverse phase chromatography. Reduction of acylpyrazines with hydrazine gave the corresponding alkylpyrazines; 3,5,6-trimethyl-2-(3-methyl-1-butyl) pyrazine and 3,5,6-Trimethyl-2-n-propylpyrazine.

(3) Sidestream aroma. Several candidate flavorants have been evaluated in an effort to alter the perception of sidestream smoke. Evaluations are conducted using E-2 conference room. Seven cigarettes are injected with the odorant and stataically

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burned in ashtrays. Panelists enter the closed room and rate overall intensity and amount of irritation on a 7-point scale. They also describe the odor. Cigarettes are extinguished and, 10 minutes later, panelists repeat the evaluation.

As expected the addition of flavorants at a high level (1500ppm) produces a distinct odor note to the smoke. However there is no significant reduction in intensity or irritation. Dose-response curves are being developed for a sub-set of these additives.

An alternative approach to the "distinctive" aroma is to develop a counteractant. In brief, by using a flavorant which blends with the smoke aroma, it may be possible to reduce intensity and irritation without delivering a "distinctive" aroma. At 160ppm, p-cresylisovalerate shows a small reduction in both intensity and irritation without a noticeable change in smoke aroma. Dose-response curves are being developed for this compound.

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PROJECT NUMBER: 2525
PROJECT TITLE: Tobacco Chemistry
PROJECT LEADER: S. Tafur
PERIOD COVERED: April, 1988

I. NATURAL PRODUCTS CHEMISTRY

A. Objective: To isolate, identify and/or analyze natural compounds with major emphasis on tobacco and tobacco products.

B. Results: In preparation for countercurrent chromatography (CCC) of a tobacco glycoside fraction, settling times were determined and partition coefficients were estimated for a number of solvent systems. Two systems provide partitioning in an acceptable range for CCC (1).

Several fractions prepared from Fr III were submitted to Flavor Development for subjective evaluation (2).

C. Plans: Continue fractionation of Bright tobacco extract fractions and purification of specific compounds.

D. References:
1. Core, M. Notebook No. 8608, p.52.
2. Tafur, S. Notebook No. 8490, p.137.

II. LOW NICOTINE

A. Objective: To examine characteristics of unextracted nicotine.

B. Results: Selected tobacco materials were submitted for nicotine analyses by both pyrolysis and extraction in an effort to understand more about the nature and occurrence of unextracted nicotine. The data indicated genetically low alkaloid tobaccos and N. glutinosa contain unextractable nicotine while tomacco (Bright tobacco grafted on tomato root stock) and Bright (Coker 347) roots do not (1).

The study of the desorption kinetics of nicotine from filler was completed. Desorption curves were plotted for neat nicotine and nicotine from control and ART fillers and rate constants were derived (2).

C. Plans: Continue to investigate genetic and cultural factors which influence the amount of unextracted nicotine in tobacco materials.

D. References:
1. Izac, R. Notebook No. 8632, p.20.
2. Core, M. Notebook No. 8608, p.52.

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III. SS/MS FLAVOR DISTRIBUTION

A. **Objective**: To examine the smoke distribution of selected flavors from flavor-release compounds.

B. **Results**: Additional PM Blues smoke samples (SS TPM and chamber wash, MS TPM, and butt) were provided to J. Naworal (Project 2501) for development of a GC/MS method for analysis of vanillin. Cigarettes were prepared with 300ppm glucovanillin core-injected or applied to the cigarette paper. Analyses of smoke fractions from these cigarettes are in progress (1).

Work was initiated with R. Levins (Project 6908) on the study of the smoke distribution of menthol from glucose menthylcarbonate (GMC) (1).

C. **Plans**: Complete both studies and report results as soon as possible.

D. **References**:
1. Tafur, S. Notebook No. 8490, p.137.

IV. GREENHOUSE STUDIES

A. **Objective**: To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.

B. **Results**: The harvest of roots from Group 10 of the hydroponic Burley 21 plants was completed. Over 11 Kg of fresh root material were produced in this group for use by Project 1904 (1).

Plans were made to set up field plots at the Whiteville, N.C. Tobacco Research Station for the production of U.S. grown Oriental tobacco under specified cultural practices (1).

C. **Plans**: Continue to produce fresh root tissue by hydroponic culture. Monitor the field plots as needed.

D. **References**:
1. Bass, R. Notebook No. 8607, pp. 21-24. (West, G. Notebook No. 8559).

V. SUPPORT ACTIVITIES

A. **Objective**: To provide requested assistance for special projects.

B. **Results**: Assistance was provided for the preparation of 65 gallons of potassium propylparaben. HPLC analyses were conducted of the prepared solution to measure the degree of hydrolysis (1).

A blended filler was sprayed with 1000 ppm β-cyclocitrylidene acetic acid (CR-2085) and handmade cigarettes were prepared (2).

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Five menthol-release compounds were sprayed onto 25g batches of a blended filler and handmade cigarettes were fabricated (3).

C. **References**:

1. Izac, R. Notebook No. 8632, p. 20.
2. Tafur, S. Notebook No. 8490, p. 137.
3. Bass, R. Notebook No. 8607, p. 21-24.

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PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: April, 1988

I. PROJECT ART

A. **Objective**: To investigate the role and fate of ammonia, carbon dioxide, and pH in the AB application for project ART.

B. **Results**: Examination of the data for the five tobacco types demonstrates different modes of ammonia and carbon dioxide loss in the various tobaccos. Bodied burley shows considerable volatile loss of ammonia over a 12 day period, while bodied bright shows no volatile loss and little if any loss due to reaction. Carbon dioxide loss demonstrated a spike ten minutes after sampling for all tobacco types. The change in pH proved to be tobacco family dependent with erratic behavior for the first hour after AB application.

C. **Conclusions**: The determination of AB in AB sprayed tobaccos can only be measured as a function of ammonia due to rapid loss of carbon dioxide. Rapid and highly variable changes are occurring in all tobacco types during the first hour after spraying which are not reflected in the ammonia determination.

D. **Plans**: To extend these and nicotine extraction studies to the DL blend.

E. **References**:

PM Notebook No. 8499.

II. ELEMENTAL ANALYSIS

A. **Objective**: To provide quantitative and qualitative elemental data on tobacco, cigarette paper, material evaluation samples, and special project samples.

B. **Results**:

K in ART Stem by XRF
Several samples of stem treated with monopotassium citrate were analyzed by XRF for potassium concentration. There was a discrepancy in comparing levels of potassium before and after treatment because of a dilution effect that was not accounted for. This problem has been resolved (1).

XRF Analysis at TQAF
Results from the TLA lab are reported on a dry weight basis. The monitor was recalibrated and the ash formula recalculated so that XRF results would be consistent with this (2). Support has been given to QA as needed during the initial startup period.

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Thin-Film Analysis by XRF

New single element thin-film standards were used to standardize the XRF spectrometer for paper analysis. Some initial cigarette and tipping paper samples have been quantitatively analyzed for Mg, Al, Si, K, Ca, Ti, and Fe. Results look promising.

Crop Samples from 1987

XRF multielement data (Mg, Si, P, S, Cl, K, and Ca) have been acquired on bright leaf, burley leaf, and bright stem of each grade and belt. A statistical evaluation will be done to determine if there is a pattern associated with belt and grade.

C. **References**:

1. Lewis, J. Y. and S. E. Wrenn, "Correction of Analytical Data for Weight Change in Processed Materials," memo to Distribution, March 30, 1988.

2. Lewis, J. Y., "Calibration of XRF Bright Monitor on a Dry Weight Basis," memo to D. Donaher, March 22, 1988.

III. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIALS

A. **Objective**: To provide headspace analyses for residual solvents from packaging materials and develop a QA method for the routine analysis of packaging materials.

B. **Results**:

1. Another gas chromatograph was put into service with an attached headspace sampler. This GC was equipped with a 60m DB-1 column and allowed samples to be analyzed in duplicate and comparisons made between the DB-1 and the poraplot Q columns. Standards were found to be stable over a period of a month when stored at ambient temperature in glass volumetric flasks capped with plastic stoppers.

2. Recovery studies of a standard solution added to a blank label paralleled the results obtained on the blank pack material. Recoveries were low and variable. Since these materials are likely to change with some frequency, the easiest approach would be to ignore matrix effects and obtain calibration runs in the usual manner, i.e. using a standard solution added to an empty glass vial.

3. Threshold values of the components of the volatiles solution were obtained. Standards were prepared at the threshold value, 10% above the threshold and 10% below the threshold. These were analyzed on the DB-1 column as well as on the poraplot Q column. The results have not as yet been evaluated. However it is evident that ethyl cellosolve is not observed at its threshold level on the DB-1 column but is seen on the poraplot column.

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C. **Plans**: Make further comparisons of the DB-1 and poraplot columns. Complete studies of threshold studies on the DB-1 and poraplot columns.

D. **References**:

1. Picone, R., "Identification of Unknown Components in Volatile Solvents," memo to R. Dunaway, March 28, 1988.

2. Picone, R., "PTS 114," memo to Rick Dunaway, April 15, 1988.

IV. RESPONSE TO ANALYTICAL REQUESTS

A. **Objective**: To provide analytical support to R&D and Operations personnel and projects.

B. **Results**:

Analyses and investigations by the project personnel during the month of April included:

Eleven brands of Japanese cigarettes were analyzed for methoprene content. No methoprene was detected.

Three customer complaint samples were analyzed this past month. No contaminants were observed.

A capillary GC procedure for methoprene analysis is being developed to replace the packed column analysis currently being used at Universal Leaf.

Volatile compounds from two adhesive samples were identified by GC/FT-IR/MS. The major volatile component from both samples was vinyl acetate.

Several different tobaccos were analyzed for nicotine by pyrolysis and solvent extraction as part of the unextracted nicotine study. These results will be reported after more experiments are completed.

C. **References**:

1. Shelton, J. H., "Methoprene/Japanese Cigarettes," memo to D. C. Watson, April 12, 1988.

2. Shelton, J. H., "Kabat Analysis/Japanese Cigarettes," memo to T. A. Newman, April 15, 1988.

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PROJECT NUMBER: 6906
PROJECT TITLE: Biological Effect of Smoke
PROJECT LEADER: J. M. Penn
WRITTEN BY: G. J. Patskan and J. M. Penn
PERIOD COVERED: April, 1988

I. PROTEIN KINASE C (PKC) WHOLE CELL ASSAY

A. **Objective**: To evaluate the effects of phorbol-12,13-dibutyrate (PDBu) and 12-O-tetradecanoyl-phorbol-13-acetate (TPA) on 3T3 cells grown at various stages.

B. **Results**: Preliminary results indicate that there was a dose-related response with both PDBu and TPA. This dose-response effect was observed in both log phase cells and after the cells were refed with 0.5% serum media. However, there does not appear to be a single peak (i.e. Mw = 90,000) of increased phosphorylation. Additional software received this month may be helpful in quantitating the previously generated autoradiographs.

C. **Plans**: Complete the evaluation of autoradiographs using the new software. Repeat the dose-response experiment with PDBu and TPA while incorporating different procedures for gel and autoradiograph exposure, development and evaluation.

D. **Reference**:

Nixon, G. Notebook No. 8569, p. 112.

II. 3T3 CELL GROWTH CONDITIONS FOR THE PKC ASSAY

A. **Objective**: To define conditions required to generate "confluent/quiescent" cells.

B. **Results**: Two experiments were performed using cells that were grown in 10% serum and were then refed daily with media containing 5% serum. Although the cell numbers remained constant after the addition of 5% serum, a significant number of dead cells were observed. This suggests that there was concurrent cell growth and cell death which may not be indicative of the quiescent stage.

C. **Plans**: Evaluate other methods of inducing and confirming the quiescent stage for 3T3 cells.

D. **Reference**:

Nixon, G. Notebook No. 8569, pp. 112.

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III. INHIBITION OF EGF BINDING ASSAY

A. **Objective**: To determine if reduced glutathione (GSH) has an effect on the activity of 2R1 IT CSC and catechol in the Inhibition of EGF Binding Assay.

B. **Results**: One experiment was conducted which examined the effects of GSH in the EGF assay. By themselves, 2R1 CSC and catechol inhibit EGF binding and reduce cell numbers. The addition of GSH (5mM) to CSC or catechol did not abrogate the effects of those agents. This preliminary data suggests that the constituents in CSC responsible for activity in the EGF assay do not react with GSH.

C. **Plans**: Repeat this experiment.

D. **Reference**:

Patskan, G. Notebook No. 8644, p. 30.

IV. PDBu BINDING ASSAY

A. **Objective**: Determine the effects of catechol on PDBu binding to 3T3 cells.

B. **Results**: In three experiments, a 19.5 hr exposure to catechol (0.01 - 10.0 µg/ml) caused a significant dose-dependent increase in the binding of ^{3}H-PDBu per cell. In two experiments, catechol elicited no significant effects when added to the cells at the time of binding.

C. **Plans**: Examine the effects of compounds that inhibit protein kinase C (PDBu receptor) activity.

D. **Reference**:

Ferguson, T. J. Notebook No. 8645, p. 45.

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PROJECT NUMBER: 6908
PROJECT TITLE: Smoke Condensate Studies
PROJECT LEADER: R. D. Kinser
PERIOD COVERED: April, 1988

I. TSNA PRECURSORS

A. **Objective**: To determine the precursors of MS TSNA.

B. **Results**: An experiment was conducted to determine if a primary amine, which does not yield a stable nitrosamine upon nitrosation, would act as a nitrosating agent scavenger and lead to reduced levels of TSNA. MS TSNA deliveries from burley base web (BuBW) + anabasine + nitrate + dodecylamine were significantly greater (~two-fold increase) for NNN, NAT, and NAB. Interestingly, NNK levels were somewhat lower. These results, in conjunction with a previous study of dodecylamine added to DBC burley and nicotine-treated low alkaloid burley, indicate that addition of a primary amine is not a viable method for TSNA reduction. However, the different response of NNK to the addition of primary amine to BuBW will be investigated in the continuing research on the precursors of MS NNK.

C. **Plans**: Determine the NNK delivery of BuBW + nitrate and BuBW + nitrate + dodecylamine.

D. **References**:

Haut, S. A. Notebook 8595, p. 66.

II. TSNA DECOMPOSITION STUDIES

A. **Objective**: To explore the thermal stability of TSNA and investigate methods to enhance thermally induced decompositions of these compounds.

B. **Results**: Initial examination of the effect of propyl dihydroxyhydrocinnamate on the thermal decomposition of NNN indicated that PrDHHC was not as effective as ascorbyl palmitate.

C. **Plans**: Replicate the PrDHHC/NNN results and determine the effect of PrDHHC on NNK decomposition.

D. **References**:

Morgan, W. R. Notebook No. 8579, p. 42.
Tickle, M. H. Notebook No. 8587, p. 130.

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III. TSNA REMOVAL STUDIES

A. **Objective**: To explore the possibility of extracting TSNA from stored tobacco using an extraction fluid compatible with current processing.

B. **Results**: The utility of Amberlyst-15 cation exchange resin for the removal of TSNA from ethanol or hexane extracts of tobacco was examined. The ion exchange chromatography was effective for removal of TSNA from a hexane extract (67 - 100% decrease) but not for removal of TSNA from the ethanol extract (12 - 29% decrease). Analyses are being conducted to determine the alkaloid removal accomplished by passage of the extract through the Amberlyst-15 column.

C. **Plans**: Complete the alkaloid analyses, and begin combining the extraction step with removal of TSNA from the extraction solvent with the Amberlyst resin.

D. **References**:

Warfield, A. H. Notebook No. 8558, p. 93.

IV. SUPPORT FUNCTION: CONDENSATE PREPARATION

A. **Objective**: To fabricate cigarettes, perform smokings, and prepare condensate as needed for biological and chemical analysis.

B. **Results**: Seventy smokings of seventeen different cigarette codes were performed to yield condensate for biological assay. A smoking machine was modified to permit condensate collection from a new model cigarette. Condensate was collected from two cigarette types in response to a request from Analytical Research.

C. **References**:

Hellams, R. D. and McGee, N. H. Notebook No. 8613, p. 54.

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PROJECT NUMBER: 0008
PROJECT TITLE: Computer Applications/General Support
PROJECT LEADER: G. D. Davis
PERIOD COVERED: March 15 - April 19, 1988

Library Palantir/Fulcrum Test

A. **Objective**: To develop a working understanding of the Palantir scanner and the Fulcrum Fultext database; to create a Fultext collection for user testing utilizing data captured through the Palantir scanner.

B. **Results**: A total of 18 user specified files were entered into the system using the Palantir Scanner. Two of these files were unable to be scanned due to smearing of the newspaper print. All the files were created from articles found in newspapers or magazines. Overall, the Palantir did an excellent job of scanning this data in. It did prove to be fairly time consuming though due to the Palantir user being a novice and the data having been derived from newspaper print.

Fulcrum was used to create a fultext database collection. It had no apparent problems with any of the files referenced. The created database and profile were brought to the Library. The necessary environment variables and path names were added to the .logins to access fultext. A demonstration/training session was held with Charity McDonald to introduce her to the basic commands, menus and terminology needed to use Fulcrum.

C. **Conclusions**: The Library's initial reaction to the fultext database seemed very positive. They were particularly impressed with the general flow of the menus and ease of use. It was determined that the Palantir scanner could not be used to enter into the system data that was generated before January of this year. Prior to this time the method of storing data was an unconcernedly cut and paste process that created shadows, lines and severe skewing of the file making it impossible for any scanner to read. However, due to improvements in this method used to store data, the Palantir scanner will be used from this date on to scan data into the system.

D. **Plans**: To remain accessible to the library users as they continue to test the Fulcrum database to answer questions or give support in anyway needed. To continue expanding the current knowledge base about the Fulcrum database and the Palantir scanner. A meeting with Fulcrum is scheduled for the week of May 9th here at CAD. At that time we will have the opportunity to ask questions, tailor screens and develop fields that will generically meet all our user requirements.

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PROJECT NUMBER: 8101
PROJECT TITLE: Cigarette Testing Services Division
SECTION LEADER: Rebecca W. Kanipe
PERIOD COVERED: April, 1988

I. MARKET ACTIVITY

A. Objective: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. Results:

1. Domestic

Philip Morris is test marketing Alpine 85 and 100 and Alpine Lights 85 and 100 cigarettes in Pittsburg, PA and Little Rock, AK. The menthol is applied to the foil, and the cigarettes are generically priced. The Alpine 85 delivers 15.4 mg tar, 1.05 mg nicotine and 0.61 mg smoke menthol; the Alpine 100 delivers 16.0 mg tar, 1.14 mg nicotine and 0.68 mg smoke menthol; the Alpine Lights 85 delivers 10.3 mg tar, 0.74 mg nicotine and 0.52 mg smoke menthol; the Alpine Lights 100 delivers 10.7 mg tar, 0.79 mg nicotine and 0.56 mg smoke menthol.

The Santa Fe Tobacco Co. is selling American Spirit 85 cigarettes (filtered and nonfiltered). These cigarettes are manufactured by G. A. Georgopulo & Co., Inc. The blend does not contain any reconstituted tobacco, expanded stems or expanded tobacco. The levels of total reducing sugars and nitrate nitrogen indicate the blend may be an all flue cured blend. The filtered cigarettes deliver 16 mg tar and 1.2 mg nicotine; the nonfiltered cigarettes deliver 27 mg tar and 1.7 mg nicotine.

Ultra-Tech Corporation of Jenkintown, Pennsylvania has introduced Smoke-Free®, a nontobacco product. This product is shaped like a cigarette and releases a sweet flavoring (saccharin) when it is touched on moist lips. This product is an 83.8 mm cellulose acetate (CA) rod wrapped with two layers of cigarette paper and has cork tipping. There are no flavorings on the CA rod; however, it does contain triacetin (9%). The package states that it is not to be lit but serves chiefly as a pacifier. A package of 3 sells for $1.00. Ultra-Tech claims that sales of this nontobacco product is in the range of $8,000 to $10,000 per week. It is being sold in 12 states.

2. International

Japan Tobacco, Inc. introduced Hi-Lite Mild Long Size cigarettes to the Japanese market on April 1. These cigarettes deliver 14 mg tar and 1.1 mg nicotine.

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II. PHYSICAL PARAMETERS OF DOMESTIC BRANDS

A. **Objective**: To issue an annual consolidated report of the physical parameters of cigarette brands including coal removal, cylinder volume, cigarette firmness, and sieve fractions.

B. **Status**: The 1987 report was issued on March 31.

C. **Results**: The data included in this report were obtained from cigarettes manufactured in 1987. Highlights of the report are given below:

1. Coal Removal: The coal removal of R. J. Reynolds Vantage 85 and Camel Lights 85 decreased during the latter half of 1987. No modifications were observed for Camel. In May, Brown and Williamson removed the expanded tobacco (11% to 0%) from the blend of Raleigh Lights 100 cigarettes. This brand showed a significant decrease in coal removal following this change.

2. Cylinder Volume: No significant changes in cylinder volume were observed for selected Philip Morris and competitive brands.

3. Cigarettes Firmness: A procedure change was made in this analysis beginning July, 1987 in order to standardize testing between Cigarette Testing Services and Quality Assurance Laboratories. Unequilibrated cigarette firmness replaced equilibrated cigarette firmness. In addition, the corresponding oven volatiles analysis was performed using unequilibrated cigarettes. The procedure change resulted in an increase in the measured cigarette firmness for all brands during the second half of 1987. This increase corresponded with lower oven volatiles of the unequilibrated cigarettes.

4. Sieve Analysis: Brown and Williamson averaged highest in the industry in the 6+12 mesh fractions, followed by Philip Morris. Philip Morris, Brown and Williamson, and American averaged lowest in the 35+ pan fractions. No significant changes in sieve fractions were noted in individual brands.

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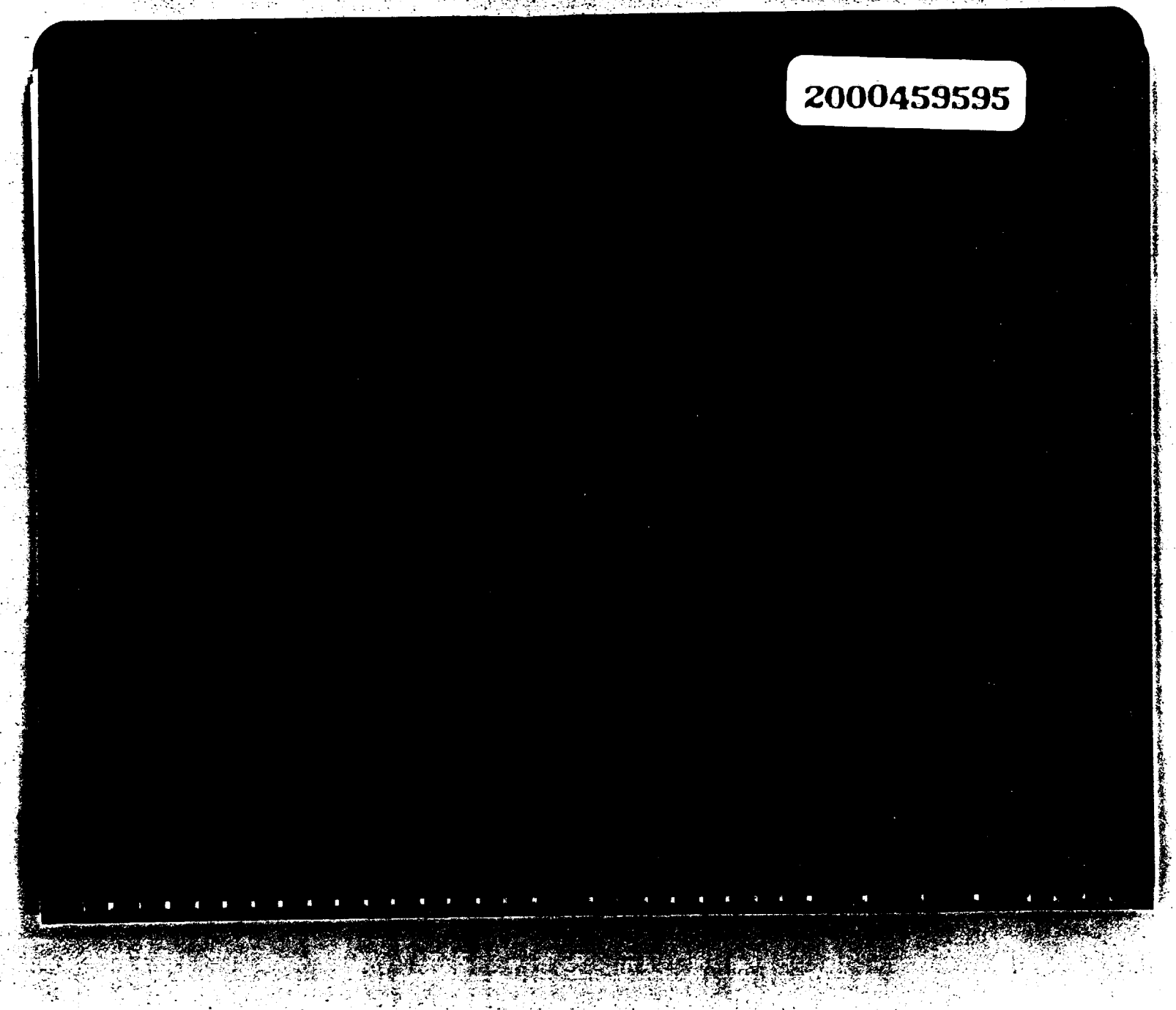

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